01334

Seneca Army Depot Activity
Romulus, New York

USACE - New York District
US Army, Engineering & Support Center
Huntsville, AL

Final UFP-QAPP

Seneca Army Depot Activity

Long-Term Monitoring

Contract No. W912DY-09-D-0062-0023
Task Order No. 0023
EPA SITE ID# NY0213820830
NY Site ID# 8-50-006

PARSONS

May 2017

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LIST OF ACRONYMS

ACRONYM	DEFINITION	ACRONYM	DEFINITION
AFFF	Aqueous Film Forming Foams	NYSDEC	New York State Department of Environmental Conservation
AOC	Area of Concern	OBOB	Open Burning/Open Burning
Ash Landfill	Ash Landfill Operable Unit	OU	Operable Unit
BRAC	Base Realignment and Closure	PAHs	Polycyclic Aromatic Hydrocarbons
CD	Compact Disk	PALPAL	Projection Action Limit
CENAN	USACE New York District	Parsons	Parsons Government Services, Inc.
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act	PDFPDF	Portable Document Format
COCCOC	Contaminant of Concern	PFOA	Perfluorooctanoic Acid
CoC	Chain of Custody	PFOS	Perfluorooctane Sulfonate
COR	Contracting Officer Representative	QC	Quality Control
CSM	Conceptual Site Model	QSM	Quality Systems Manual
cy	Cubic Yards	RA	Remedial Action
DA	Department of the Army	RCRA	Resource Conservation and Recovery Act
DFW	Definable Feature of Work	RDR	Remedial Design Report
DL	Detection Limit	ROD	Record of Decision
DOD	Department of Defense	ROM	Read-Only Format
DQO	Data Quality Objective	SCIDA	Seneca County Industrial Development Agency
DUR	Data Usability Report	SDG	Sample Delivery Group
ft	Feet	SEAD-16	Abandoned Deactivation Furnace
LCS	Laboratory Control Sample	SEAD-17	Active Deactivation Furnace
LIMS	Laboratory Information Management System	SEAD-25	Fire Training and Demonstration Pad
LOD	Limit of Detection	SEDA	Seneca Army Depot Activity
LOQ	Limit of Quantitation	SOPs	Standard Operating Procedures
LTM	Long-Term Monitoring	SVOCs	Semi-Volatile Organic Compounds
LUCs	Land Use Controls	TAL	Target Analyte List
MCL	Maximum Contaminant Level	TOC	Total Organic Carbon
MEE	Methane, Ethane, Ethene	TPP	Technical Project Planning
MPCs	Measurement Performance Criteria	UFP-QAPP	Uniform Federal Policy – Quality Assurance Project Plan
MS	Matrix Spike	U.S.	United States
MSD	Matrix Spike Duplicate	USACE	U.S. Army Corps of Engineers
NCFL	Non-Combustible Fill Landfill	USAEHA	U.S. Army Environmental Hygiene Agency
NCP	National Contingency Plan	USAESCH	U.S. Army Engineering Support Center, Huntsville
NGVD	National Geodetic Vertical Datum	USEPA	United States Environmental Protection Agency
NYS	New York State	VOCs	Volatile Organic Compounds

Executive Summary

ES.1 Introduction

The United States (U.S.) Army Engineering and Support Center, Huntsville (USAESCH) has retained Parsons Government Services, Inc. (Parsons) to continue and maintain the Remedial Action (RA) at various sites per the Record of Decision (ROD). Parsons will perform this work consistent with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and the National Contingency Plan (NCP), 40 CFR Part 300. All activities involving work in areas potentially containing explosive hazards shall be conducted in full compliance with the United States Army Corps of Engineers (USACE), Department of the Army (DA), and Department of Defense (DoD) regulations, guidance, standards, and manuals.

The Seneca Army Depot Activity (SEDA) is a 10,587-acre former military facility located in Seneca County in the towns of Varick and Romulus, New York, and was owned by the United States Government and operated by the Department of the Army between 1941 and 2000. In 2000, the Army closed the Depot and assumed a caretakers' role of the property, pending the closeout of its continuing environmental obligations and the leasing or transfer of property to other public or private parties for beneficial reuse purposes. Since 2000, approximately 9,250 acres of land have been transferred to other parties.

This Uniform Federal Policy – Quality Assurance Project Plan (UFP-QAPP) describes the methods and procedures necessary to complete the long-term monitoring (LTM) and achieve the required project objectives for the following sites at SEDA:

- Open Burning (OB) Grounds;
- Fire Training and Demonstration Pad (SEAD-25);
- Ash Landfill Operable Unit (Ash Landfill); and
- Abandoned Deactivation Furnace (SEAD-16) and Active Deactivation Furnace (SEAD-17)

The UFP-QAPP also contains additional methods and procedures which are applicable to the sampling of emerging contaminants (perfluroalkyl substances [PFAS] also known as perfluorooctanoic acid [PFOA] and perfluorooctane sulfonate [PFOS]) at the following sites where Aqueous Film Forming Foams (AFFF) (e.g., firefighting foams) may have been used:

- Fire Training and Demonstration Pad (SEAD-25);
- Fire Training Pit (SEAD-26); and
- Airfield and Fuel Pads (SEAD-122E)

A brief overview of each of the seven sites listed is provided below.

ES.1.1 OB GROUNDS

The OB Grounds, located in the northwestern portion of SEDA, was used for demilitarization of munitions for approximately forty years. A RA was conducted between 1999 and 2004 to address the potential exposure to elevated levels of metals (i.e., lead and copper) detected in the site soils and the sediments located in the adjacent Reeder Creek. The remedy specified in the ROD (Parsons, 1999a) included removal of the berms surrounding the historic burn pads; removal of all soils to a depth of at least 1 foot; placement of a 9-inch vegetative cover over any soils with lead concentrations greater than 60 mg/kg, but less than or equal to 500 mg/kg; excavation of sediments in Reeder Creek with elevated levels of copper or lead; and implementation of a monitoring program for groundwater, sediment, and the capped areas. LTM activities have been conducted since 2007 in accordance with the LTM Plan (Parsons, 2007a) at the OB Grounds to assess groundwater, vegetative soil cap, and Reeder Creek conditions. The observations and findings from the latest round of LTM at the OB Grounds is presented in the 2015 LTM Annual Report (Parsons, 2016b).

ES.1.2 SEAD-25

SEAD-25, located in the east-central portion of SEDA, was in use from the late 1960s to the late 1980s. The former pad was used for fire control training, including fire-fighting demonstrations. In accordance with the ROD (Parsons, 2004) and the Final Remedial Design Work Plan and Design Report (Parsons, 2005), a RA was completed in 2005 and removed approximately 1,722 cubic yards (cy) of soil and sediment impacted by volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) at SEAD-25 as documented in the Final Construction Completion Report (Parsons, 2006). Since groundwater concentrations were found exceeding the applicable groundwater standards prior to the RA, LTM activities have been carried out at SEAD-25 since 2006 following the RA. Additionally, SEAD-25 has been inspected to ensure that established Land Use Controls (LUCs) are enforced. The LUCs in place at SEAD-25 include: prohibit the development and use of property for residential housing, elementary and secondary schools, childcare facilities, and playgrounds; and prohibit access to or use the groundwater, other than for monitoring purposes, until the applicable New York State Department of Environmental Conservation (NYSDEC) Class GA groundwater standards are met. The observations and findings from the latest round of LTM at SEAD-25 is presented in the 2015 Annual LTM Report (Parsons, 2015).

ES.1.3 ASH LANDFILL

The Ash Landfill, located in the west-central portion of SEDA, was used from 1941 to 1974 to burn uncontaminated trash in a series of burn pits located near the former abandoned incinerator building (Building 2207). According to the U.S. Army Environmental Hygiene Agency (USAEHA) Interim Final Report, Groundwater Contamination Survey No. 38-26-0868-88 (USAEHA, 1987), the ash from the refuse burning pits was buried in the Ash Landfill (SEAD-6) from date of inception until the late 1950s or early 1960s. Other areas of the site were used as a grease pit and for burning debris. Post-closure, the landfill was covered with native soil, but was not closed with an engineered cover or cap.

In 2006, in accordance with the ROD (Parsons, 2004), the Remedial Design Work Plan (Parsons, 2006b) and the Remedial Design Report (RDR) (Parsons, 2006c), a RA was completed. The RA involved the following: Installation of three dual biowall systems, to address volatile organic compounds (VOCs) in groundwater that exceed New York State (NYS) Class GA groundwater standards; construction and establishment of a 12-inch vegetative cover over the Ash Landfill and the Non-Combustible Fill Landfill (NCFL) to prevent ecological receptors from coming into direct contact with underlying soils that are contaminated with metals and polycyclic aromatic hydrocarbons (PAHs); excavation and disposal of three debris piles; and re-grading of the incinerator cooling water pond to promote positive drainage. LTM activities have been conducted since 2007 at the Ash Landfill to assess groundwater conditions, the effectiveness of the biowall, and the vegetative soil cap. The observations and findings from the latest round of LTM at the Ash Landfill is presented in the Annual Report and Year 9 Review (Parsons, 2016c).

ES.1.4 SEAD-16 AND SEAD-17

SEAD-16 and SEAD-17 are located in the east-central portion of the SEDA within the former ammunition storage area. SEAD-16, the former Abandoned Deactivation Furnace, was used from approximately 1945 until the mid-1960s when its use ceased and the site was vacated. SEAD-17, the former Active Deactivation Furnace, was constructed to replace the Abandoned Deactivation Furnace at SEAD-16. However, SEAD-17 was inactive after 1989 as a result of Resource Conservation and Recovery Act (RCRA) permitting issues. Based on historic site activities, soils from both sites were contaminated with select metals (i.e., antimony, arsenic, cadmium, copper, lead, mercury, thallium, and zinc) at levels above identified risk-based action levels. Additionally, soils at SEAD-16 were also contaminated with PAHs. The RA implemented at SEAD-16/17 (Parsons, 2008) involved the removal of 4,427 cy of impacted soil. The ROD (Parsons, 2006) also required the implementation, maintenance, inspection, and periodic reporting of LUCs to prohibit the use of the land for residential purposes and restrict access and use of groundwater until applicable cleanup standards are achieved. The long-term groundwater monitoring has been performed at SEAD-16/17 in accordance with the ROD and the Final Work Plan (Parsons, 2007b) since 2007. The observations and findings from the latest round of LTM at the SEAD-16/17 is presented in the Annual Report 2015 – Year 8 (Parsons, 2016d).

ES.1.5 SEAD-26

SEAD-26 is located in the southeastern portion of SEDA and was used one to four times a year for firefighting training during which time various flammable materials were floated on water, ignited, and extinguished. Prior to 1977, the fire training area may have also been used for fire demonstrations. In accordance with the ROD (Parsons, 2004) and the Final Remedial Design Work Plan and Design Report (Parsons, 2005), a RA was completed in 2005 and removed approximately 828 cubic yards of soil impacted with carcinogenic polycyclic aromatic hydrocarbons (cPAHs) (Parsons, 2006). Prior to the RA, groundwater at SEAD-26 was found to be impacted by VOCs; however, a significant plume was not found. Three rounds of semi-annual LTM was conducted at SEAD-26. No contaminants of concern (COCs) were detected in any of the rounds therefore, with concurrence from NYSDEC, LTM at this site was concluded (Parsons, 2007c).

ES.1.6 SEAD-122E

SEAD-122E, and the surrounding airfield, are located in the southwest corner of SEDA. SEAD-122E is associated with the deicing of planes at three separate aircraft refueling areas at the former SEDA Airfield. All three of the historic deicing/refueling pads that comprise SEAD-122E are located along the western side of the northwest-southeast runway. Two of the deicing/refueling pads are located near either end of the runway, while the third is located at the end of a short taxiway, west of the central portion of the runway. An Environmental Baseline Survey was conducted to investigate the three pads and determined if they were impacted by deicing fluids used on planes (Parsons, 1999b). SVOCs, mainly PAHs and phathalates, were detected in soil; none exceeded screening criteria. Groundwater was not found to be impacted (Parsons, 2007).

ES.2 Project Objectives and Technical Approach

The project objective is to continue the LTM program in order to monitor groundwater conditions until they are below the performance criteria standards and to monitor that the remedy continues to be effective. Once this has been accomplished at a particular site, the U.S. Environmental Protection Agency (USEPA) and NYSDEC may approve of the termination of long-term groundwater monitoring, LUCs, or both. Additionally, PFAS will be sampled at SEADs-25, -26, and 122E. The Conceptual Site Model (CSM) for each site is described on **Worksheet #10**.

Project-specific data quality objectives (DQOs) were developed based on this CSM and these are described on **Worksheet #11** of this UFP-QAPP. These DQOs include a design for obtaining data to support the LTM at each site and a design for sampling emerging contaminants related to AFFF. The design for obtaining data described in the last column of the DQO tables on **Worksheet #11** summarizes the technical approach. The project approach is described in detail on **Worksheet #17**, and specific analyzes are noted on **Worksheet #18**. The primary components of the long-term groundwater sampling design for the LTM at each of the sites involve collecting samples to be analyzed using low-flow techniques. The general scope of the ongoing LTM activities for each of the sites are as follows:

- OB Grounds
  - Collect groundwater samples and record geochemical parameters from 6 existing monitoring wells;
  - Analyze samples for total copper and total lead;
  - Inspect the vegetative cap for disturbances;
  - Inspect Reeder Creek for evidence of soil transport from OB Grounds; and
  - Confirm LUCs are in compliance.
- SEAD-25
  - Collect groundwater samples and record geochemical parameters from 5 existing monitoring wells;
  - Analyze samples for VOCs, Methane, Ethane, Ethene (MEE), chloride, sulfate, sulfide, iron, sodium, nitrate and nitrite; and
  - Confirm LUCs are in compliance.
- Ash Landfill
  - Collect groundwater samples and record geochemical parameters from 14 existing monitoring wells;

  - Analyze samples for VOCs, MEE, sulfate, manganese, ferrous iron, and total organic carbon (TOC);
  - Determine effectiveness of biowall and whether recharge is required; and
  - Inspect the vegetative cap for disturbances.
- SEAD-16/17
  - Collect groundwater samples and record geochemical parameters from 11 existing monitoring wells; and
  - Analyze samples for target analyte list (TAL) metals; and
  - Confirm LUCs are in compliance.

The general scope of the activities related to sampling for PFAS at each of the sites are as follows:

- SEAD-26 and SEAD-122E
  - Collect groundwater grab samples using direct push drilling techniques to install temporary well points and collect samples from the wells; and
  - Analyze samples for PFAS.
- SEAD-25
  - Collect groundwater grab samples using existing 12 monitoring wells; and
  - Analyze samples for PFAS.

While these components are the focus of the project, the field operations involve multiple elements, or "definable features of work" (DFWs) that will be required to achieve the project goals. These DFWs are listed on **Worksheet #14** and they are explained further in this worksheet, with references to relevant standard operating procedures (SOPs) (**Worksheet #21** and **Appendix A and D**), measurement performance criteria (MPCs) (**Worksheet #12**), and other sections of the UFP-QAPP, as necessary.

ES.3 Document Organization

This UFP-QAPP was prepared under Task Order 0023 of Contract W912DY-09-D-0062, in accordance with UFP-QAPP, Optimized UFP-QAPP Worksheets (EPA, 2012), EPA QA/G-5 (EPA, 2002), and EM 200-1-15 to ensure environmental data collected are scientifically sound, of known and documented quality, and suitable for their intended purposes. This UFP-QAPP focuses on the site-specific details for the LTM and sampling of PFAS at the OB Grounds, SEAD-25, SEAD-26, the Ash Landfill, SEAD-122E, and SEAD-16/17 to include monitoring methods, analytical services, data management and validation procedures, and field and laboratory SOPs.

This UFP-QAPP presents the plan for collecting data to support the LTM and PFAS sampling and uses the "optimized" worksheets format published by the Intergovernmental Data Quality Task Force in March 2012 (EPA, 2012). Supporting plans and other information are included in the references section of this UFP-QAPP.

Crosswalk from UFP-QAPP Manual to Worksheets

This UFP-QAPP presents the plan for collecting data to support the LTM at the OB Grounds, SEAD 25, Ash Landfill, and SEAD 16/17 and PFAS sampling at the SEAD 25, SEAD 26, and SEAD 122 sites at the Seneca Army Depot Activity, and "optimized" worksheets published by the Intergovernmental Data Quality Task Force in March 2012. The optimized worksheets address all requirements of ANSI/ASQ E4-2004 and CIO 2106-G-05. The following table provides a "crosswalk" between the worksheets and the respective elements of CIO 2106-G-05. In addition, each revised worksheet includes a reference to the appropriate CIO 2106-G-05 element.

OPTIMIZED UFP-QAPP WORKSHEETS		2106-G-05 QAPP GUIDANCE SECTION	
1&2	Title and Approval Page	2.2.1	Title, Version, and Approval / Sign-Off
3&5	Distribution List and Project Organization	2.2.3 2.2.4	Distribution List Project Organization and Schedule
4, 7 & 8	Personnel Qualifications and Sign-Off Sheet	2.2.1 2.2.7	Title, Version, and Approval / Sign-Off Special Training Requirements and Certification
6	Communication Pathways and Procedures	2.2.4	Project Organization and Schedule
9	Project Planning Session Summary	2.2.5	Project Background, Overview, and Intended Use of Data
10	Conceptual Site Model	2.2.5	Project Background, Overview, and Intended Use of Data
11	Data Quality Objectives	2.2.6	Data/Project Quality Objectives and Measurement Performance Criteria
12	Measurement Performance Criteria	2.2.6	Data/Project Quality Objectives and Measurement Performance Criteria
13	Secondary Data Uses and Limitations	Chapter 3	QAPP Elements for Evaluating Existing Data
14 & 16	Project Tasks & Schedule	2.2.4	Project Organization and Schedule
15	Project Action Limits and Laboratory-Specific Detection / Quantitation Limits	2.2.6	Data/Project Quality Objectives and Measurement Performance Criteria
17	Sampling Design and Rationale	2.3.1	Sample Collection Procedure, Experimental Design, and Sampling Tasks
18	Sampling Locations and Methods	2.3.1 2.3.2	Sample Collection Procedure, Experimental Design, and Sampling Tasks Sampling Procedures and Requirements
19 & 30	Sample Containers, Preservation, and Hold Times	2.3.2	Sampling Procedures and Requirements
20	Field Quality Control	2.3.5	Quality Control Requirements
21	Field Standard Operating Procedures	2.3.2	Sampling Procedures and Requirements
22	Field Equipment Calibration, Maintenance, Testing, and Inspection	2.3.6	Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables
23	Analytical Standard Operating Procedures	2.3.4	Analytical Methods Requirements and Task Description
24	Analytical Instrument Calibration	2.3.6	Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection	2.3.6	Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables
26 & 27	Sample Handling, Custody, and Disposal	2.3.3	Sample Handling, Custody Procedures, and Documentation

OPTIMIZED UFP-QAPP WORKSHEETS		2106-G-05 QAPP GUIDANCE SECTION	
28	Analytical Quality Control and Corrective Action	2.3.5	Quality Control Requirements
29	Project Documents and Records	2.2.8	Documentation and Records Requirements
31, 32 & 33	Assessments and Corrective Action	2.4	Assessments and Data Review
		2.5.5	Reports to Management
34	Data Verification and Validation Inputs	2.5.1	Data Verification and Validation Targets and Methods
35	Data Verification Procedures	2.5.1	Data Verification and Validation Targets and Methods
36	Data Validation Procedures	2.5.1	Data Verification and Validation Targets and Methods
37	Usability Assessment	2.5.2	Quantitative and Qualitative Evaluations of Usability
		2.5.3	Potential Limitations on Data Interpretation
		2.5.4	Reconciliation with Project Requirements

Worksheets #1 & 2: Title and Approval Page

(EPA UFP-QAPP Guidance Manual, Section 2.1; EPA Guidance 2106-G-05 Section 2.2.1)

1.1 PROJECT IDENTIFYING INFORMATION

Site Name / Project Name:	Seneca Army Depot Activity / Remedial Action
Site Location / No.:	Romulus, NY, EPA Site ID# NY0213820830, NY Site ID# 8-50-006
Contract / TO No.:	W912DY-09-D-0062 / Task Order 0023

1.2 CONCURRING SIGNATURES

The below signatures indicate the representatives of the subject organizations have reviewed this UFP-QAPP and concur with its implementation as written.

Lead Organization / Contracting Officer Representative

POMMERENCK.DEREK.ANDREW.1080769748

Digitally signed by POMMERENCK.DEREK.ANDREW.1080769748
DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USA, cn=POMMERENCK.DEREK.ANDREW.1080769748
Date: 2017.06.13 08:19:43 -05'00'

Derek Pommerenck, USAESCH Project Manager — Date

Lead Organization / Project Manager

BATTAGLIA.RANDALL.W.1228816724

Digitally signed by BATTAGLIA.RANDALL.W.1228816724
DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USA, cn=BATTAGLIA.RANDALL.W.1228816724
Date: 2017.06.13 09:12:18 -04'00'

Randy Battaglia, USACE New York District (CENAN) — Date

Contractor Project Manager

Beth Badik — May 23, 2017

Beth Badik, Parsons Project Manager — Date

Federal Regulatory Agency

Julio Vazquez, USEPA Regional Project Manager — Date

State Regulatory Agency

Melissa Sweet, NYSDEC Project Manager — Date

Contractor Quality Assurance

Tammy Chang — May 30, 2017

Tammy Chang, Parsons Quality Manager — Date

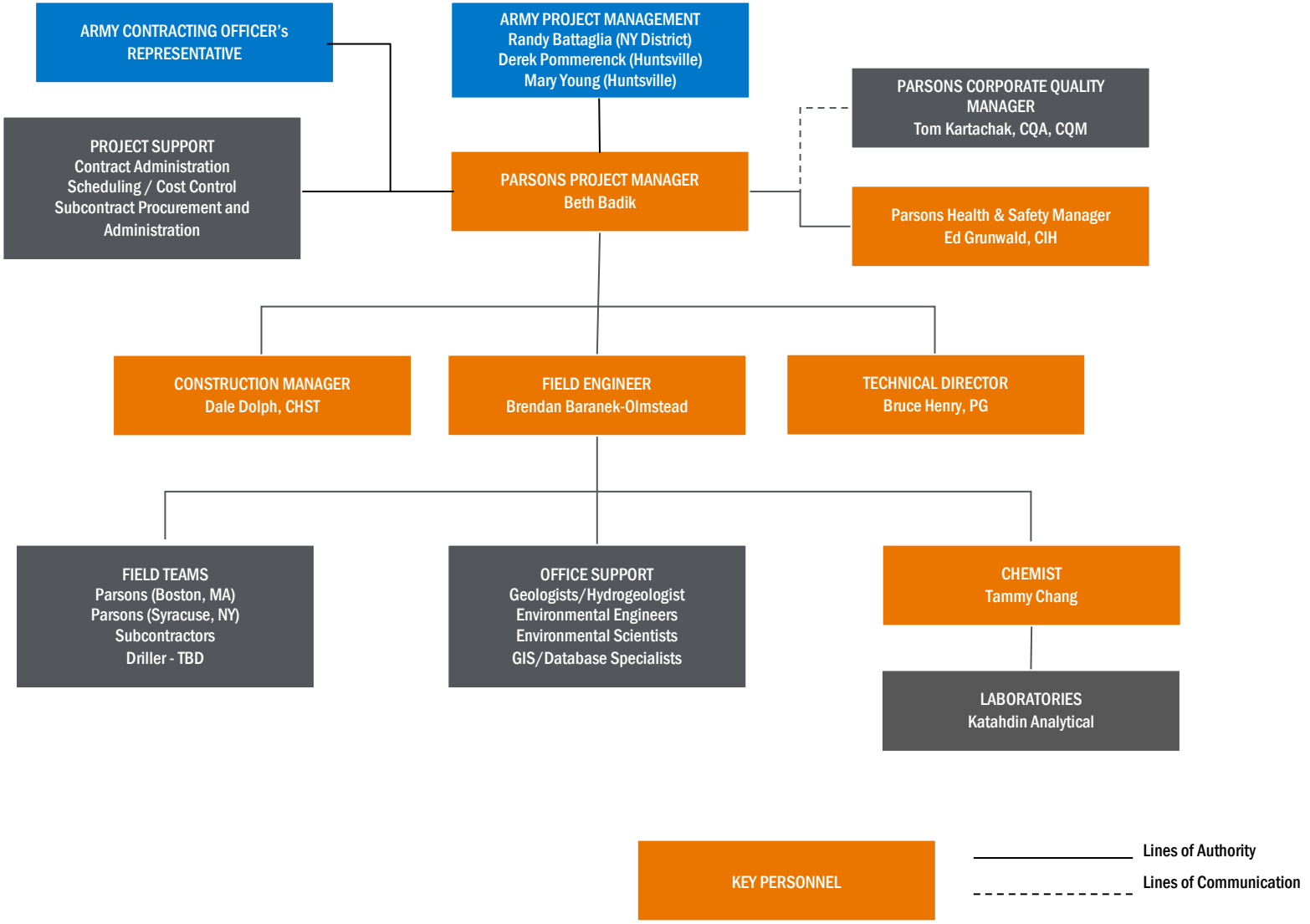
1.3 QAPP IDENTIFYING INFORMATION

Guidance Used:	Uniform Federal Policy for Quality Assurance Project Plans, Optimized UFP-QAPP Worksheets (USEPA, 2012); EPA QA/G-5 (EPA, 2002); and EM 200-1-15
Regulatory Program:	Base Realignment and Closure (BRAC), CERCLA
Approval Entity:	USAESCH
Data Users:	U.S. Army, USEPA, NYSDEC
QAPP Type:	Optimized UFP- QAPP
Scoping Sessions	See **Worksheet #9**
Previous UFP-QAPPs:	None

Worksheets #3 & 5: Project Organization and QAPP Distribution

(EPA UFP-QAPP Guidance Manual, Section 2.4.1; EPA Guidance QA/G-5, Sections 2.1.3 and 2.2.4)

FIGURE 3.1 – PROJECT ORGANIZATION AND QAPP DISTRIBUTION



Worksheets #4, 7, & 8: Personnel Qualifications and Sign-Off Sheet

(EPA UFP-QAPP Guidance Manual, Section 2.4.3, EPA Guidance QA/G-5, Section 2.1.8)

4.1 KEY PROJECT PERSONNEL

PROJECT TITLE/ROLE	NAME/ ORGANIZATION	CONTACT INFORMATION (TELEPHONE/E-MAIL)	EXPERIENCE	SPECIALIZED TRAINING/ CERTIFICATIONS	SIGNATURE/DATE (1)
USACE Contracting Officer Representative (COR)	Derek Pommerenck USAESCH	256-895-1794 Derek.Pommerenck@usace.army.mil	n/a	n/a	*Signature on Worksheets #1 & 2*
USACE Project Manager (PM)	Randy Battaglia CENAN	607-869-1523 Randy.W.Battaglia@usace.army.mil	n/a	n/a	*Signature on Worksheets #1 & 2*
Contractor PM	Beth Badik Parsons	617-449-1565 beth.badik@parsons.com	Over 10 years of experience as PM conducting HTRW investigations	BS, Chemical Engineering, 2001	*Signature on Worksheets #1 & 2*
Federal Regulator	Julio Vazquez USEPA Region 2	212-637-4323 Vazquez.Julio@epa.gov	n/a	n/a	*Signature on Worksheets #1 & 2*
State Regulator	Melissa Sweet NYSDEC	518-402-9614 melissa.sweet@dec.ny.gov	n/a	n/a	*Signature on Worksheets #1 & 2*
Quality Assurance Manager	Tammy Chang Parsons	512-719-6092 Tammy.Chang@parsons.com	Over 25 years of analytical laboratory and chemistry-related experience	MS, Chemistry, 1977	*Signature on Worksheets #1 & 2*

(1) Signatures indicate personnel have read this UFP-QAPP and agree to implement the procedures as written.

4.2 OTHER PROJECT PERSONNEL

PROJECT TITLE/ROLE	NAME/ ORGANIZATION	CONTACT INFORMATION (TELEPHONE/E-MAIL)	EXPERIENCE	SPECIALIZED TRAINING/ CERTIFICATIONS (1)	RECEIVES COPY OF QAPP
USACE PM	Derek Pommerenck USAESCH	256-895-1794 Derek.Pommerenck@usace.army.mil	n/a	n/a	Yes
USACE Technical Manager	Brett Frazier USAESCH	256-895-1874 Brett.W.Frazier@usace.army.mil	n/a	n/a	Yes
Environmental Health Risk Assessor	Keith Hoddinott APHC	410-436-5209 Keith.B.Hoddinott.civ@mail.mil	n/a	n/a	Yes
Contractor Construction Manager	Dale Dolph Parsons	315-506-3939 Dale.Dolph@parsons.com	Over 20 years of experience in the sampling of hazardous materials and substances related to hazardous waste site investigations, petroleum storage facilities, and site assessments. Also includes construction management/oversight for a variety of environmental remediation.		No
Contractor Technical Director	Bruce Henry Parsons	(303) 831-8100x1986 Bruce.Henry@parsons.com	Over twenty years of experience providing geological and engineering services for hazardous waste remediation and petroleum exploration.	M.S. Geology, 1993 B.S. Geology, 1981	No
Contractor Field Engineer	Brendan Baranek-Olmstead Parsons	617-449-1404 brendan.baranek-olmstead@parsons.com	Over 11 years of environmental consulting, field work oversight, and various field sampling methods	MS, Environmental Engineering, 2004; BS, Civil Engineering, 2002	No
Contractor Corporate Quality Manager	Tom Kartachak Parsons	410-596-9178 Tom.Kartachak@parsons.com	Over 39 year of experience, including 19 years of experience ensuring effective implementation of planning programs and projects, including Quality Manager	M.S., Public Health, 1977; B.S., Biology, 1974	Yes
Contractor Health & Safety Manager	Ed Grunwald Parsons	678-969-2394 Ed.Grunwald@parsons.com	31 years of experience developing and implementing safety programs for environmental remediation, Military Munitions Response Program (MMRP) and construction projects	Certified Industrial Hygienist	No
Contractor Project Chemist	Tammy Chang Parsons	512-719-6092 Tammy.Chang@parsons.com	Over 25 years of analytical laboratory and chemistry-related experience	MS, Chemistry, 1977	Yes
Analytical Laboratory Project Manager	David Alltucker TestAmerica- W. Sacramento	916-374-4383 david.alltucker@testamericainc.com	Over 18 years of analytical laboratory and chemistry-related experience	B.A., Chemistry	No
Contractor Data Validator	Maryanne Kosciewicz Parsons	315-552-9703 Maryanne.Kosciewicz@parsons.com	Over 20 years of experience with data review, evaluation, and validation.	B.S, Mathematics B.S. Chemistry	Yes
Analytical Laboratory QA Officer	Lisa Stafford TestAmerica- W. Sacramento	916-374-4308 lisa.stafford@testamericainc.com	Over 28 years of analytical laboratory and chemistry-related experience	B.S. Chemistry, 1986	

PROJECT TITLE/ROLE	NAME/ ORGANIZATION	CONTACT INFORMATION (TELEPHONE/E-MAIL)	EXPERIENCE	SPECIALIZED TRAINING/ CERTIFICATIONS (1)	RECEIVES COPY OF QAPP
Analytical Laboratory Project Manager	Heather Manz Katahdin Analytical Services	207-874-2400x17 hmanzjobrin@katahdinlab.com	15 years of analytical laboratory and chemistry-related experience with Katahdin	B.S. Ocean Studies, 1999	Yes
Analytical Laboratory QA Officer	Leslie Dimond Katahdin Analytical Services	207-874-2400 ext. 20 ldimond@katahdinlab.com	22 years of analytical laboratory and chemistry-related experience with Katahdin	B.A., Chemistry	Yes

Worksheet #6: Communication Pathways and Procedures

(EPA UFP-QAPP Guidance Manual, Section 2.4.2, EPA Guidance 2106-G-05 Section 2.2.4)

COMMUNICATOR DRIVER	INITIATOR (ROLE)[1][2]	RECIPIENT(S) (ROLE)[1]	PROCEDURE
General communication between Lead Organization and other process development team (PDT) members	Lead Organization PM or designee	Appropriate PDT member(s)	Communicates directly as needed (verbally and/or in writing).
Regulatory interface	Lead Organization PM	Regulator	Provides project update via e-mail at least every other week during field activities.
Regulatory oversight	Regulator	Lead Organization PM	Communicates directly as needed (verbally and/or in writing).
Project management, Task Order administration and logistics, QAPP changes prior to field/laboratory work	Contractor PM	Lead Organization PM and/or lead technical and site management personnel	Communicates directly as needed (verbally and/or in writing).
Weekly project conference calls	Contractor PM	Lead Organization PM, appropriate PDT member(s)	Communicates project status verbally via weekly conference call.
Field progress reports	Contractor Field Engineer	Contractor PM and lead technical and site personnel. Serves as Field Team Leader.	Documents progress in daily report and submits to Contractor PM for onward distribution to PDT. Daily reports will be submitted to USAESCH PM within 24 hrs of work completion that day whenever possible.
Stop work due to safety issues	Contractor Field Engineer	Contractor PM	Verbally notify Contractor PM as soon as possible after work stoppage.
	Contractor PM	Lead Organization and Design Center PMs	Notify USAESCH PMs verbally or via e-mail as soon as possible after work stoppage.
QAPP changes in the field	Contractor Field Engineer	Contractor PM	Follows change and review and approval process; Communicates directly as needed (verbally and/or in writing) and submits draft Field Change Request form for discussion; does not implement change until approval is granted; consults with other personnel as needed.
	Contractor PM	Lead Organization and Design Center PMs State and Federal Regulators	Submits Field Change Request form to USAESCH for approval; does not implement change until approval is granted. State and Federal regulators notified of significant changes to the QAPP via email or phone during the field event. Will not implement until approval granted.
Field corrective actions	Contractor Field Engineer	Contractor PM	The need for field corrective actions will be determined by the Contractor PM, and/or contractor technical personnel. The contractor technical personnel will notify the Contractor PM of any needed field corrective actions and the Contractor PM will respond within 24 hrs.
	Contractor PM	State and Federal Regulators	Field corrective actions will be included in the site-specific report.
Reporting laboratory Quality Control (QC) variances or sample receipt variances	Analytical or QA Laboratory Project Manager	Contractor Project Chemist	Applicable Laboratory Project Manager will notify Contractor Project Chemist verbally and in writing. All sample receipt variances will be communicated within 24 hrs of sample receipt.
	Contractor Project Chemist	Contractor PM	Contractor Project Chemist will notify Contractor PM immediately for significant variances.

COMMUNICATOR DRIVER	INITIATOR (ROLE)(1)(2)	RECIPIENT(S) (ROLE)(1)	PROCEDURE
Analytical corrective actions and data validation corrective actions	Contractor Project Chemist	Contractor PM	The need for Corrective Actions for analytical and data validation issues will be determined by the Contractor Project Chemist and laboratory when error occurs during the analysis or noticed during data review or validation stage. Corrective action report will be included in the associated data package.
	Contractor PM	USAESCH PMs, and Technical Manager	The Contractor PM will notify USAESCH of any non-conformance lab issues if errors cause rejected data or re-analysis cannot be performed due to holding time.
	Contractor PM	State and Federal Regulators	Any non-conformance lab issues will be documented in the site-specific report.
Reporting data validation issues	Contractor Project Chemist Contractor Data Validator	Analytical or QA Laboratory Project Manager	All completeness and data issues will be addressed with the laboratory directly, verbally and in writing immediately in case the team is still in the field and samples can be recollected. The validated data package will be due within approximately 14 calendar days of receipt. Data validator will validate laboratory data package with analytical results.

(1) Names and contact information for personnel provided on **Worksheets #4, 7,** & **8**.

(2) The initiator may designate another qualified individual to communicate with the recipient(s); however, the initiator shown is responsible for the communication being made.

Worksheet #9: Project Planning Session Summary

(EPA UFP-QAPP Guidance Manual, Section 2.5.1 and Figures 9-12, EPA Guidance 2106-G-05 Section 2.2.5)

Technical Project Planning (TPP) meetings have not been held to discuss the LTM at Seneca. However, weekly telephone conferences are held between project management to discuss the relevant action items and project planning related to upcoming activities to be performed at Seneca. The primary focus of the conference calls are to discuss upcoming field activities, project deliverables, and document reviews. A list of the weekly conference call participants is included in the table below.

NAME	ORGANIZATION	TITLE / ROLE	E-MAIL / PHONE
Randy Battaglia	CENAN	Seneca AD BRAC Environmental Coordinator/Caretaker	Randy.W.Battaglia@usace.army.mil 607-869-1523
Derek Pommerenck	USAESCH	Project Manager	Derek.Pommerenck@usace.army.mil 256-895-1794
Mary Young	USAESCH	Technical Manager	Mary.K.Young@usace.army.mil 256-895-1874
Keith Hoddinott	USACHPPM	Environmental Health Risk Assessor	Keith.B.Hoddinott.civ@mail.mil 410-436-5209
Beth Badik	Parsons	Project Manager	Beth.Badik@parsons.com 617-449-1565

Worksheet #10: Conceptual Site Model

(EPA UFP-QAPP Guidance Manual, Section 2.5.2, EPA Guidance 2106-G-05 Section 2.2.5)

10.1 OVERVIEW

The primary purpose of this worksheet is to describe the CSMs for each of the project sites. In order to provide the basis for this, this worksheet also summarizes observations from previous investigations, information from site reports, on-going LTM, details of the contaminants and the affected matrices, and other relevant supporting information. Further details for each site are available in the respective LTM reports.

10.2 SITE DESCRIPTION AND BACKGROUND

SEDA, a 10,587-acre former military facility located in Seneca County near Romulus, New York, is located between Seneca Lake and Cayuga Lake in Seneca County, and is bordered by New York State Highway 96 to the east, New York State Highway 96A to the west (**Figure 10.1**), and sparsely populated farmland to the north and south. The facility was wholly owned by the United States Government and was operated by the Department of the Army between 1941 and 2000; since 2000, portions of the Depot have been transferred to other parties for reuse. The primary mission of SEDA was the receipt, storage, maintenance, and supply of military items. A location map of the LTM sites at SEDA is presented as **Figure 10.2**.

10.2.1 SITE LOCATION

OB Grounds

The OB Grounds is located in the northwestern portion of the Depot where the planned future use of the land currently is designated for conservation purposes**.** As situated, the OB Grounds sits a minimum of 1,780 feet away from the nearest Depot boundary, which is located to the west of the area of concern (AOC) (**Figure 10.2**). The OB Grounds site sits on gently sloping terrain and is bounded on the east by Reeder Creek, a perennial creek that is generally less than 1 foot deep and which eventually flows into Seneca Lake (**Figure 10.3**). The quality of surface water in Reeder Creek is designated by the State of New York as a Class C water body (best usage of fresh water is fishing; the waters shall be suitable for fish propagation and survival). Seneca Lake is located approximately 10,000 feet west of the OB Grounds site and is used as a source of drinking water for numerous surrounding communities and the Depot.

The OB Grounds is vegetated with grass and brush and there are no permanent structures within the area other than small concrete bunkers and a metal garage structure. The former Open Detonation Area (SEAD-45) is located immediately north of the OB Grounds, and the former Explosive Ordnance Disposal Area (SEAD-57) is located approximately 4,000 to 5,000 feet south of the former OB Grounds. The OB Grounds was historically used for surface burning of explosive trash and propellants.

SEAD-25

The Fire Training and Demonstration Pad (SEAD-25) is located in the east-central portion of SEDA. The site is bounded to the east by Administration Avenue, beyond which is undeveloped land covered by deciduous trees; to the south by Ordnance Drive beyond which is an open grassy field and a stand of coniferous trees; to the west by a drainage ditch running from the northeast to the southwest with grassland, brush and conifers between the site and the ditch; and, to the north by grassland and a former baseball field. A site map of the SEAD-25 area and its location within the SEDA is included as **Figure 10.1**. As situated, SEAD-25 sits a minimum of 1,350 feet away from the nearest SEDA boundary, which is located to the east of the AOC. A more detailed site map of SEAD-25 is provided as **Figure 10.4**. SEAD-25 was in

use from the late 1960s to the late 1980s. The former pad was used for fire control training. During the 1980s, the pad was used twice for fire-fighting demonstrations, including one demonstration in 1982 or 1983, and one in 1987.

Ash Landfill

The Ash Landfill OU, also referred to as the Ash Landfill, is located in the west-central portion of SEDA (**Figure 10.1**) where vehicular and pedestrian access is restricted. The Ash Landfill is composed of five areas of concern (AOCs). The five AOCs that comprise the Ash Landfill Operable Unit (OU) are the Incinerator Cooling Water Pond (SEAD-3), the Ash Landfill (SEAD-6), the NCFL (SEAD-8), the former Debris Piles (SEAD-14), and the former Abandoned Solid Waste Incinerator Building (SEAD-15) (**Figure 10.5**).

SEAD-16/17

SEAD-16 and SEAD-17 are located in the east-central portion of the SEDA within the former ammunition storage area in an area where vehicular and pedestrian access is restricted. SEAD-16 and SEAD-17 are located in the portion of SEDA where land is presently designated for future PID uses. The locations of SEAD-16 and SEAD-17 are shown in **Figure 10.6 and 10.7**.

SEAD-122E

SEAD-122E, and the surrounding airfield, are located in the southwest corner of SEDA. Three of the four of the historic deicing/refueling pads that comprise SEAD-122E are located along the western side of the northwest-southeast runway, and the fourth fuel pad is located to the east near the central portion of the runway. Two of the deicing/refueling pads are located near either end of the runway, a third is located at the end of a short taxiway to the west of the central portion of the runway, and a fourth to the east of the central portion of the runway (**Figure 10.9**).

SEAD-26

The Fire Training Pit (SEAD-26) site is located in the southeastern portion of SEDA (**Figure 10.10**). The site is bounded to the east and west by SEDA railroad tracks; on the south by grassland and low brush; and on the north by 7th Street. Vehicular access is provided to the site via a locking gate on 7th Street.

10.2.2 TOPOGRAPHY AND VEGETATION

SEDA is located in an uplands area, where the elevation ranges from approximately 600 feet (ft.) National Geodetic Vertical Datum (NGVD 1929) along the western boundary of the Depot to nearly 760 feet NGVD 1929 in the central portion of the eastern boundary. The uplands area where SEDA is located forms a divide separating two of the New York Finger Lakes: Cayuga Lake on the east and Seneca Lake on the west. Sparsely populated farmland covers most of the surrounding area. In general, the Ash Landfill and OB Grounds sites are located on the western side of the topographic divide and SEADs 16, 17, and 25 are on the eastern side.

Vegetation across the Depot consists of successional old field, successional shrub, and successional hardwoods.

10.2.3 GEOLOGY, SITE SOILS, AND WILDLIFE

The Finger Lakes uplands area is underlain by a broad north-to-south trending series of rock terraces mantled by glacial till. As part of the Appalachian Plateau, the region is underlain by a tectonically undisturbed sequence of Paleozoic rocks consisting of shale, sandstone, conglomerate, limestone, and dolostone. In the vicinity of SEDA, Devonian age (approximately 385 million years ago) rocks of the Hamilton Group are monoclinally folded and dip gently to the south. The Hamilton Group is a sequence of limestone, calcareous shale, siltstone, and sandstone.

SEDA geology is characterized by gray Devonian shale with a thin weathered zone where it contacts the overlying mantle of Pleistocene glacial till. This stratigraphy is consistent over the entire SEDA facility. The predominant surficial geologic

unit present at the site is dense glacial till. The till is distributed across the entire facility and ranges in thickness from less than 2 feet to as much as 15 feet although it is generally only a few feet thick. The till is generally characterized by brown to gray-brown silt, clay and fine sand with few fine-to-coarse gravel-sized inclusions of weathered shale. Larger diameter weathered shale clasts (as large as 6-inches in diameter) are more prevalent in basal portions of the till.

The bedrock underlying the Site is composed of the Ludlowville Formation of the Devonian age, Hamilton Group. Regionally, the bedrock is vertically jointed in three predominant directions: northeast, north-northwest, and east-northeast (Mozola, 1951; Merin, 1992). The Hamilton Group is a gray-black, calcareous shale that is fissile and exhibits parting (or separation) along bedding planes.

Pleistocene age (Wisconsin event, 20,000 years ago) glacial till deposits overlies the shale. SEDA lies on the western edge of a large glacial till plain between Seneca Lake and Cayuga Lake. The till matrix, the result of glaciations, varies locally but generally consists of horizons of unsorted silt, clay, sand, and gravel. The soils at SEDA contain varying amounts of inorganic clays, inorganic silts, and silty sands. In the central and eastern portions of SEDA, the till is thin and bedrock is exposed or within 3 feet of the surface. The thickness of the glacial till deposits at SEDA generally ranges from 1 to 15 feet.

Darien silt-loam soils, 0 to 18 inches thick, have developed over Wisconsin age glacial tills. These soils are developed on glacial till where they overlie the shale. In general, the topographic relief associated with these soils is from 3 to 8 percent (%).

10.2.4 GROUNDWATER AND SURFACE WATER CONDITIONS

Regionally, four distinct hydrologic units were identified within Seneca County (Mozola, 1951). These include two distinct shale formations, a series of limestone units, and unconsolidated beds of Pleistocene glacial drift. Overall, the groundwater in the county is very hard, and therefore, the quality is minimally acceptable for use as potable water.

Regionally, the water table aquifer of the unconsolidated surficial glacial deposits of the region would be expected to flow in a direction consistent with the ground surface elevations. Geologic cross-sections from Seneca Lake and Cayuga Lake were constructed by the State of New York (Mozola, 1951, and Crain, 1974). The geologic cross-sections suggest that a groundwater divide exists approximately half way between the two Finger Lakes. SEDA is located on the western slope of this divide and therefore regional groundwater flow is expected to be primarily westward towards Seneca Lake. Local hydrogeology is overall consistent with the regional hydrogeology.

Surface drainage from SEDA flows to five primary creeks. In the southern portion of the Depot, the surface drainage flows through man-made drainage ditches and streams into Indian and Silver Creeks. These creeks then merge and flow into Seneca Lake just south of the SEDA airfield. The central part and the administration area of the SEDA drain into Kendaia Creek. Kendaia Creek flows in a predominant westerly direction and discharges into Seneca Lake at a location north of Pontius Point and the SEDA former Lake Shore Housing Area. The majority of the northwestern and north-central portion of the SEDA drains into Reeder Creek. Reeder Creek flows predominantly northwesterly and leaves the Depot at a point that is north of the Open Detonation Area (i.e., SEAD-45) and west of the former Weapons Storage Area or the “Q” before it turns to the west and flows into Seneca Lake. The northeastern portion of the Depot, which includes a marshy area called the Duck Pond, drains into Kendig Creek and then flows north into the Cayuga-Seneca Canal and to Cayuga Lake. Other minor creeks are also present and drain portions of the Depot.

10.3 HISTORY

10.3.1 OB GROUNDS

The land at the OB Grounds was used for demilitarization of munitions for approximately forty years. The open burning procedure involved the preparation of combustible beds of pallets and wooden boxes on the pads followed by the placement of ammunition or the components to be demilitarized on the beds. A trail of propellant was placed on the

ground leading to the combustible bed. Once ignited the energetic material was allowed to burn until only ash and casing residues remained. Items burned included various military munitions such as propellants and projectiles.

The burning of munitions has been performed at designated burning pads, which range in size from approximately 100 by 100 feet to 300 by 800 feet. There were a total of nine (9) such pads at the OB Grounds. The burning pads at the site are built on top of the natural glacial till soils. Originally, demilitarization of munitions was performed via open burning on the ground surface. Difficulties in sustaining the burning process were noted due to the poor drainage characteristics of the soil. Subsequently, individual burn pads were built up with crushed shale and soils to provide a drier environment in which to perform the burning. Each burn pad has from 1/2 to 2 feet of crushed shale at the surface. Below this material are the pre-existing agricultural soils overlying the glacial till. Berms surround each of the burning pads on three sides.

Designated munition waste was open-burned on the nine separate burning pads until 1987. After 1987, munitions were destroyed by burning them within an aboveground steel tray to minimize the impact of the burning on the environment.

10.3.2 SEAD-25

SEAD-25 was in use from the late 1960s to the late 1980s. The former pad was used for fire control training. During the 1980s, the pad was used twice for fire-fighting demonstrations, including one demonstration in 1982 or 1983, and one in 1987.

10.3.3 ASH LANDFILL

Prior to the Army's purchase of land for construction of the SEDA, the area of the Ash Landfill OU was used for farming. From 1941 (the date SEDA was constructed) to 1974, uncontaminated trash was burned in a series of burn pits located near the former abandoned incinerator building (Building 2207). According to the U.S. Army Environmental Hygiene Agency (USAEHA) Interim Final Report, Groundwater Contamination Survey No. 38-26-0868-88 (July 1987), the ash from the refuse burning pits was buried in the Ash Landfill (SEAD-6) from date of inception until the late 1950s or early 1960s.

The incinerator was built in 1974. Between 1974 and 1979, materials intended for disposal were transported to the incinerator. Each week the Depot generated approximately 18 tons of refuse, the majority of which was incinerated. The source for the refuse was domestic waste from Depot activities and family housing. Large items that could not be burned were disposed at the NCFL (SEAD-8). The NCFL encompasses approximately three acres located southeast of the former incinerator building, immediately south of a SEDA railroad line. The NCFL was used as a disposal site for non-combustible materials, including construction debris, from 1969 until 1977.

Ash and other residue from the former incinerator were temporarily disposed in an unlined cooling pond immediately north of the incinerator building. The cooling pond consisted of an unlined depression approximately 50 feet in diameter and approximately 6 to 8 feet deep. When the pond filled, the fly ash and residues were removed, transported, and buried in the adjacent ash landfill east of the cooling pond. The refuse was dumped in piles and occasionally spread and compacted. No daily or final cover was applied during operation. According to an undated aerial photograph of the incinerator during operation, the active area of the Ash Landfill extended at least 500 feet north of the incinerator building, near a bend in a dirt road. A fire destroyed the incinerator on May 8, 1979, and the landfill was subsequently closed. Post-closure, the landfill was apparently covered with native soil of various thicknesses, but was not closed with an engineered cover or cap. Other areas at the site were used as a grease pit and for burning debris.

10.3.4 SEAD-16/17

SEAD-16, the former Abandoned Deactivation Furnace, was used from approximately 1945 until the mid-1960s when its use ceased and the site was vacated. The site consisted of 2.6 acres of fenced land with grasslands in the north, east, and west; a storage area for empty boxes and wooden debris located to the west; and an unpaved roadway in the south. Building S-311, which previously housed the deactivation furnace, was located at the approximate center of this area, and was demolished as part of the RA at SEAD-16. Documentation of demolition activities is presented in the *Building Cleaning and Building Demolition Completion Report* (Parsons, 2008). Building S-366, known as the Process Support

Building, is located to the northeast of former Building S-311, and is currently unused and vacant. In addition to Building S-366, two sets of SEDA railroad tracks and utilities are presently on-site.

SEAD-17, the former Active Deactivation Furnace, was constructed to replace the Abandoned Deactivation Furnace at SEAD-16. However, SEAD-17 was inactive after 1989 as a result of RCRA permitting issues. SEAD-17 formerly consisted of the deactivation furnace, associated air pollution control equipment, and a support building (Building S-367), which were demolished or dismantled during the RA. Details and results of the demolition are documented in the *Building Cleaning and Building Demolition Completion Report* (Parsons, 2008). The former SEAD-17 deactivation furnace facility and support building were surrounded by a crushed shale road, beyond which lie grasslands. An unpaved gravel road to the north permits vehicular access to SEAD-17.

10.3.5 SEAD-122E

SEAD-122E is associated with the deicing of planes at three separate aircraft refueling areas at the former SEDA Airfield. The property where the airfield currently sits was once part of the Sampson Naval Training Station which was open from 1942 to 1946, and which was used for basic training of naval personnel. An Environmental Baseline Survey was conducted to investigate the three pads and determined if they were impacted by deicing fluids used on plane. SVOCs, mainly PAHs and phathalates, were detected in soil; none exceeded screening criteria. Groundwater was not found to be impacted.

10.3.6 SEAD-26

SEAD-26 is located in the southeastern portion of SEDA and was used between 1977 to 1994. The site was used one to four times a year for firefighting training during which time various flammable materials were floated on water, ignited, and extinguished. Prior to 1977, the fire training area may have also been used for fire demonstrations. In accordance with the ROD (Parsons, 2004) and the Final Remedial Design Work Plan and Design Report, a RA was completed in 2005 and removed approximately 828 cubic yards of soil impacted with carcinogenic polycyclic aromatic hydrocarbons (cPAHs). Prior to the RA, groundwater at SEAD-26 was found to be impacted by VOCs; however, a significant plume was not found. Beginning in 2007, three rounds of semi-annual LTM was conducted at SEAD-26. No contaminants of concern (COCs) were detected in any of the rounds therefore, with concurrence from NYSDEC, LTM at this site was concluded.

10.4 CURRENT AND PROJECTED LAND USE

To address employment and economic impacts associated with the closure of SEDA, the Seneca County Board of Supervisors established the Seneca Army Depot Local Redevelopment Authority (LRA) in October 1995. The primary responsibility assigned to the LRA was to prepare a plan for redevelopment of the SEDA property. Following a comprehensive planning process, a Reuse Plan and Implementation Strategy for Seneca Army Depot was completed and adopted by the LRA on October 8, 1996. The Seneca County Board of Supervisors subsequently approved this Reuse Plan on October 22, 1996. In 2005, after it had acquired land at the former Depot from the Army, the Seneca County Industrial Development Agency (SCIDA) revised the planned use designations of land in many portions of the former Depot. **Figure 10.8** depicts the intended future land uses for SEDA, as modified by the SCIDA. Since 1995, approximately 9,250 acres of the former Depot has been released to the SCIDA and other parties.

10.4.1 OB GROUNDS

LTM is an integral component of the approved remedy implemented at the OB Grounds. The ROD, Former Open Burning Grounds Site, Final" (Parsons, 1999a) indicated that monitoring of groundwater and the vegetated soil cover at the OB Grounds, and of the sediment within Reeder Creek was required. In accordance with the approved remedy as presented in the ROD, the current LTM activities at the Site per the LTM Monitoring Plan for the OB Grounds (Parsons, 2007a) include the annual collection and analysis of groundwater samples for lead and copper concentrations; the inspection of the vegetated, compacted soil cover; and, the inspection of Reeder Creek where the Creek abuts the OB Grounds.

10.4.2 SEAD-25

Currently, SEAD-25 is part of a groundwater long-term monitoring program and the land is not being used. SEAD-25 is part of the PID/Warehousing Area and the planned future use for this tract of land is for industrial, office development, and/or warehouse areas.

10.4.3 ASH LANDFILL

The Ash Landfill as part of the "PID Retained Parcels" was transferred to the SCIDA with a Quitclaim Deed executed on May 27, 2011. The Ash Landfill was transferred with the land use restrictions, consistent with the LUC Objectives as defined in the LUC RD. The deed for the PID/Warehousing Area incorporated by reference the land use restrictions set forth in the Environmental Easement.

As the selected remedies do not allow unrestricted use and unlimited exposures, the Army or its successors are required to complete a review of the selected remedies at least once every five years, in accordance with Section 121(c) of the CERCLA. The selected LUC remedy is reviewed in accordance with this inspection frequency; the LUCs are inspected as part of the FYR and on an annual basis.

10.4.4 SEAD-16/17

Currently, SEAD-16/17 is part of a groundwater long-term monitoring program and the land is not being used. SEAD-16/17 is part of the PID/Warehousing Area and the planned future use for this tract of land is for industrial, office development, and/or warehouse areas.

10.4.5 SEAD-122E

The property was active from 1942 until it was officially closed in 2000, but is currently utilized by the New York State Police for training and special events. Future use of the site is for Industrial and County Fire Training.

10.4.6 SEAD-26

SEAD-26 was in use from 1977 to 1994. An action was required at SEAD-26 to ensure land use remains protective of site users. SEAD-26 is part of the PID/Warehouse Area and the planned future use for this tract of land is for industrial, office development, and/or warehouse areas.

10.5 PREVIOUS INVESTIGATIONS

10.5.1 OB GROUNDS

The remedy specified in the ROD for the OB Grounds included removal of the berms surrounding the historic burn pads; the removal of all soils to a depth of at least 1 foot; the placement of a 9-inch vegetative cover over any soils with lead concentrations greater than 60 mg/kg, but less than or equal to 500 mg/kg; the excavation of sediments in Reeder Creek with elevated levels of copper or lead; and the implementation of a monitoring program for groundwater, sediment, and the capped areas. The first four of these required remedial actions were conducted between June 1999 and May 2004 by Weston Solutions Inc.

Currently, the LTM component of the remedy is being implemented by Parsons. LTM began in November 2007 and 10 sampling events have been completed; the most recent event was conducted in October 2015. LTM at the OB Grounds site was initially scheduled to occur on a quarterly basis. The results of the first four LTM rounds were combined and summarized in an annual report, in which, the recommended frequency of monitoring was recommended to change from quarterly to annually. Based on comments received from EPA and NYSDEC in 2009, the Army authorized the

performance of an inspection of Reeder Creek. The monitoring frequency of groundwater was agreed upon by EPA and NYSDEC in February 2010 to be conducted annually. Subsequent to Round 5, investigations at the OB Grounds have included yearly groundwater sampling and inspection of both the soil caps and Reeder Creek.

Long-term monitoring activities include the collection of groundwater quality data to monitor the effectiveness of the implemented remedy at the Site for preventing future impacts to groundwater at the OB Grounds and to sediments in Reeder Creek. Additionally, monitoring of the vegetated compacted soil cover placed over the contaminated soils at the OB Grounds is required to assure the long-term integrity of the soil cover, including the potential mobilization and migration of lead-contaminated soil buried beneath the cover; and to prevent direct contact with, and incidental ingestion of, soils containing lead at concentrations up to 500 mg/kg by terrestrial wildlife at the Site. Part of the OB Grounds LTM program includes a qualitative assessment (i.e., visual inspection) of Reeder Creek for evidence of migration of material via surface water flow or groundwater transport of contaminants into the remediated section of Reeder Creek adjacent to and down gradient of the OB Grounds. COCs continue to remain below applicable screening criteria. LTM will continue until closure is negotiated between the Army and the regulators.

10.5.2 SEAD-25

Excavation of BTEX-impacted soil at SEAD-25 pad was completed in December 2005. Soil removal totaled approximately 961 cy. The depth of excavation extended to the top of the competent shale bedrock, or approximately 4.5 feet bgs. Confirmatory soil samples collected showed that that site-specific cleanup goals were achieved and the Army determined that soils at SEAD-25 did not require further action. The EPA and NYSDEC concurred with this determination that the excavation of the soil at the pad removed the source of groundwater contamination.

Excavation of the SVOC-impacted soil in the swale at SEAD-25 was completed in November 2005. The soil excavation extended to bedrock from the toe of slope on one bank to the toe of slope on the other bank, resulting in the removal and off-site disposal of approximately 761 cy of soil from SEAD-25. After the excavation, the swale bottom consisted of exposed competent bedrock, and since no native overburden soil remained in the swale, no confirmatory samples were collected or analyzed.

A total of approximately 1,722 cy (approximately 2,600 tons) of soil were excavated from the pad and the swale at SEAD-25 and disposed off-site at Ontario County Landfill. The pad excavation was backfilled with approximately 793 cy of on-site fill material and 168 cy of fill material obtained from an off-site source, and restored to the existing grade.

LTM began in January 2006 and 13 sampling events have been completed; the most recent event was conducted in March 2016. Semi-annual groundwater monitoring of the ten monitoring wells (MW25-2, MW25-3, MW25-8, MW25-9, MW25-10, MW25-13, MW25-15, MW25-17, MW25-18, and MW25-19) located at SEAD-25 continued through 2013. The EPA and NYSDEC agreed, as recommended in the SEAD-25 *Fourth Long-Term Monitoring and Site Review Report* (Parsons, 2011c) and *Draft Final Five-Year Review Report* (Parsons, 2011d), to reduce the frequency of the semi-annual monitoring events to annual monitoring events. It was also agreed to reduce the number of wells to be monitored from ten to five since the down-gradient wells have shown no contaminants of concern (COCs) during any of the post-RA sampling events. Beginning in 2014, the focus of the sampling effort is on wells MW25-2, MW25-3, MW25-9, MW25-10 and MW25-17 where historic information indicates that COCs of interest were detected. As of the most recent LTM report, groundwater contamination was restricted to the area around MW25-2 with COC concentrations at, or below, applicable groundwater standards (Parsons, 2016a).

10.5.3 ASH LANDFILL

Prior to the listing of SEDA on the NPL, two removal actions were performed at the Ash Landfill. The first action was the removal of a former 1000-gallon underground storage tank (UST) that was used to store heating oil and was located on the east side of the abandoned Incinerator Building. The second, a Non-Time Critical Removal Action (NTCRA), was conducted by the Army in 1994/1995 and consisted of the excavation and thermal treatment of soil impacted with VOCs (Parsons, 2005c).

As part of a demonstration study, a 650-foot long permeable reactive iron wall (zero valent iron [ZVI]) was installed near the western property line of the Ash Landfill AOC (ETI, 2001). A pilot study was performed by Parsons and the Army from July 2005 to February 2006 to show that the use of mulch as the selected wall medium (i.e. biowalls) would effectively control migration of groundwater contaminants at the site. The components and findings of the mulch biowall pilot study, which serve as the basis of design for the biowalls is presented in the "Evaluation Report for the Mulch Biowalls at the Ash Landfill" submitted as an appendix of the "Draft Remedial Design Work Plan for the Ash Landfill Operable Unit" (Parsons, 2006a,b).

Since a wall material other than iron was selected, the Army conducted a review of the remedy's effectiveness one year after the walls are installed. Subsequent annual reviews were performed until the first FYR.

The first four rounds of groundwater sampling were performed in the first year of LTM and were completed in January 2007, March 2007, June 2007, and November 2007. As part of the Year 1 report, the Army recommended that the frequency of LTM events at the Ash Landfill OU be reduced from quarterly to semi-annually; this recommendation was approved by the USEPA and NYSDEC. Ten years of groundwater monitoring and 21 sampling events have been completed; the most recent sampling event was conducted in June 2016.

10.5.4 SEAD-16-17

The selected remedy for SEAD-16 and SEAD-17 included excavation of soil impacted with metals and PAHs at concentrations greater than the site-specific cleanup standards. The excavation of the impacted soil took place in July and August 2007. Approximately 1,862 cy of impacted soil was removed from SEAD-16 and approximately 2,565 cy of impacted soil was removed from SEAD-17.

Soil was excavated from both SEAD-16 and SEAD-17 until confirmatory soil samples collected from the sidewalls (when appropriate), the excavation floor, and the perimeter yielded analytical results below site-specific cleanup standards. The depth of excavation completed at SEAD-16 varied from approximately 1 to 3 feet below ground surface (bgs) and the excavation depth at SEAD-17 varied from approximately 1 to 2 feet bgs. The impacted soil from SEAD-16 and SEAD-17 was transported off-site and was disposed as non-hazardous material.

Deeper excavations at SEAD-16 and SEAD-17, including excavation areas surrounding the railroad tracks, were backfilled with clean bank-run gravel. SEAD-16 and SEAD-17 were graded to promote positive drainage. The areas at SEAD-17 that were vegetated prior to the RA were seeded to restore the vegetation. SEAD-16 was not seeded since it was not previously vegetated.

LTM began in December 2007 and eight rounds of annual sampling have been conducted. The most recent event was completed in December 2015. No LTM sampling event was conducted in 2011 due to budgetary constraints.

10.5.5 SEAD-122E

In response to a request by EPA, the Army presented the results of a risk assessment in a memo submitted in March 2005. The cancer and non-cancer risks for all future potential receptors (industrial worker, construction worker, day care center – worker, and day care center – child) and exposure routes (inhalation of dust in air, ingestion of soil or groundwater, or dermal contact to soil) for SEAD-122E were evaluated. An unacceptable cancer risk was found due to dermal contact to soil and ingestion of soil. The contributing COCs are carcinogenic PAHs in soils. For comparison purposes, risk to residential receptors was evaluated. The non-cancer HIs were less than 1.0. Land use controls include a restriction on the development and use of property for residential housing, elementary or secondary schools, child care facilities, and playgrounds until unrestricted use and unlimited exposure criteria are attained within the AOC.

10.5.6 SEAD-26

At SEAD-26, the primary contaminants detected included SVOCs and metals in the soil and sediments. In addition, low levels of volatiles were also detected in the groundwater at levels above NYSDEC GA Standards. However, the

contaminants that exceeded NYSDEC GA Standards in the groundwater were no longer found in the soil of SEAD-26 due to attenuation of the contaminants in the soil (Parsons ES, 1998). The initial excavation at SEAD-26 began on November 9, 2005 and was completed on November 15, 2005. Five distinct areas at SEAD-26 were excavated to a depth of 1 foot bgs, and a total of 828 cubic yards (1,248 tons) of soil was excavated and disposed off-site. Confirmatory soil samples were collected from the perimeter and the base of each of the five excavation areas and were analyzed for cPAHs. The edges of the five excavation areas were smoothed. All confirmatory samples representative of soil remaining on-site met the soil cleanup goals. Additional remediation of soils at SEAD-26 was not required.

LTM was conducted beginning in 2007; however, groundwater monitoring at SEAD-26 was terminated by the Army, with the approval of the USEPA and the NYSDEC, after the first year of sampling and analysis indicated that no COCs were present in the groundwater at concentrations above defined cleanup goals.

10.6 CONCEPTUAL SITE MODEL

The CSM is a description of a site and its environment that can be used to depict the nature of potential contamination, its location, and the possible interactions of human and environmental receptors with that contamination. The CSM summarizes which potential receptor exposure pathways are (or may be) complete and which are (and are likely to remain) incomplete. An exposure pathway is considered incomplete unless *all four* of the following elements are present (USEPA, 1989):

- A source of contamination;
- An environmental transport and/or exposure medium;
- A point of exposure at which the contaminant can interact with a receptor; and
- A receptor and a likely route of exposure at the exposure point.

If any single factor was not present, the pathway would be incomplete. An incomplete exposure pathway indicates there are no current means by which a receptor (human or ecological) can be exposed. In this case no hazards or risks from exposure would be expected. This information can be used to focus the investigation of the site by suggesting which complete or potentially complete exposure pathways need to be evaluated. The CSM is a 'living document' that is based on existing knowledge and, therefore, can and should be updated throughout the course of the project as more data become available.

For the purposes of this investigation, a CSM was developed for each site for which LTM is on-going. The CSM is summarized in **Tables 10.1 through 10.6.** These tables describe the known or suspected contamination sources, potential/suspected location and distribution of contamination, contamination source or exposure medium, current and future receptors, and potentially complete exposure pathways. The CSM will be revised based on investigation results and Army and stakeholder feedback.

Except SEAD-122E, surface and subsurface soil pathways are incomplete at each site because the source was either removed (SEADs 16, 17, 25, and 26) or a combination of removal and burial under a soil cover (OB Grounds). Surface and subsurface soil pathways are complete at SEAD-122E because the source was never removed. Surface water and sediment pathways are incomplete at SEADs 16, 17, 25, 26, and 122E because the media do not exist. Currently, surface water and sediment pathways are incomplete at OB Grounds because there is no evidence of erosion of on-site soils to the creek. Periodic monitoring of the soil cover and creek is conducted to assess whether evidence of erosion or protective cover breaching are present. Although five of the six sites (not including OB Grounds) have land use controls which restrict groundwater use, exposure to groundwater through ingestion is considered potentially complete for current and future on-site workers, current and future off-site residents, and future residents if groundwater is accessed.

See **Worksheet #17** for additional discussion on COCs.

Table 10.1 - Overview of Preliminary Conceptual Site Model, OB Grounds, Seneca Army Depot Activity

SITE DETAILS	KNOWN OR SUSPECTED CONTAMINATION SOURCE(S)	POTENTIAL/SUSPECTED LOCATION AND DISTRIBUTION	SOURCE OR EXPOSURE MEDIUM	CURRENT AND FUTURE RECEPTORS	POTENTIALLY COMPLETE EXPOSURE PATHWAY
NAME: OB Grounds **Acreage**: 30 acres **Release mechanisms**: Demilitarization of munitions was performed via open burning on the ground surface **Current and Future Land Use**: No current use. Proposed future use is conservation/recreation.	Metals (copper and lead)	Potentially present in groundwater as a result of leaching of the interred soil.	Groundwater	Current and future onsite worker, off-site residential. Future on-site residential.	Exposure to groundwater (ingestion)

Table 10.2 - Overview of Preliminary Conceptual Site Model, SEAD-25, Seneca Army Depot Activity

SITE DETAILS	KNOWN OR SUSPECTED CONTAMINATION SOURCE(S)	POTENTIAL/SUSPECTED LOCATION AND DISTRIBUTION	SOURCE OR EXPOSURE MEDIUM	CURRENT AND FUTURE RECEPTORS	POTENTIALLY COMPLETE EXPOSURE PATHWAY
NAME: SEAD-25 **Acreage**: 8 acres **Release mechanisms**: Former fire control training. Release of petroleum products. **Current and Future Land Use**: No current use. Future use is planned industrial development.	VOCs (primarily BTEX) PFAS	Potentially present in groundwater. Former soil source was removed prior to LTM.	Groundwater	Current and future onsite worker, off-site residential. Future on-site residential.	Exposure to groundwater (ingestion)

Table 10.3 - Overview of Preliminary Conceptual Site Model, Ash Landfill, Seneca Army Depot Activity

SITE DETAILS	KNOWN OR SUSPECTED CONTAMINATION SOURCE(S)	POTENTIAL/SUSPECTED LOCATION AND DISTRIBUTION	SOURCE OR EXPOSURE MEDIUM	CURRENT AND FUTURE RECEPTORS	POTENTIALLY COMPLETE EXPOSURE PATHWAY
NAME: Ash Landfill **Acreage**: 45 acres **Release mechanisms**: Former use of incinerator and burial of ash. **Current and Future Land Use**: No current use. Future use is planned industrial development.	VOCs (predominantly chlorinated VOCs)	Potentially present in groundwater. Former soil source was removed prior to LTM.	Groundwater	Current and future onsite worker, off-site residential. Future on-site residential.	Exposure to groundwater (ingestion)

Table 10.4 - Overview of Preliminary Conceptual Site Model, SEAD-16/17, Seneca Army Depot Activity

SITE DETAILS	KNOWN OR SUSPECTED CONTAMINATION SOURCE(S)	POTENTIAL/SUSPECTED LOCATION AND DISTRIBUTION	SOURCE OR EXPOSURE MEDIUM	CURRENT AND FUTURE RECEPTORS	POTENTIALLY COMPLETE EXPOSURE PATHWAY
NAME: SEAD-16/17 **Acreage**: 13 acres **Release mechanisms:** Demilitarization of munitions **Current and Future Land Use**: No current use. Future use is planned industrial development.	TAL Metals	Potentially present in groundwater. Former soil source was removed prior to LTM.	Groundwater	Current and future onsite worker, off-site residential. Future on-site residential.	Exposure to groundwater (ingestion)

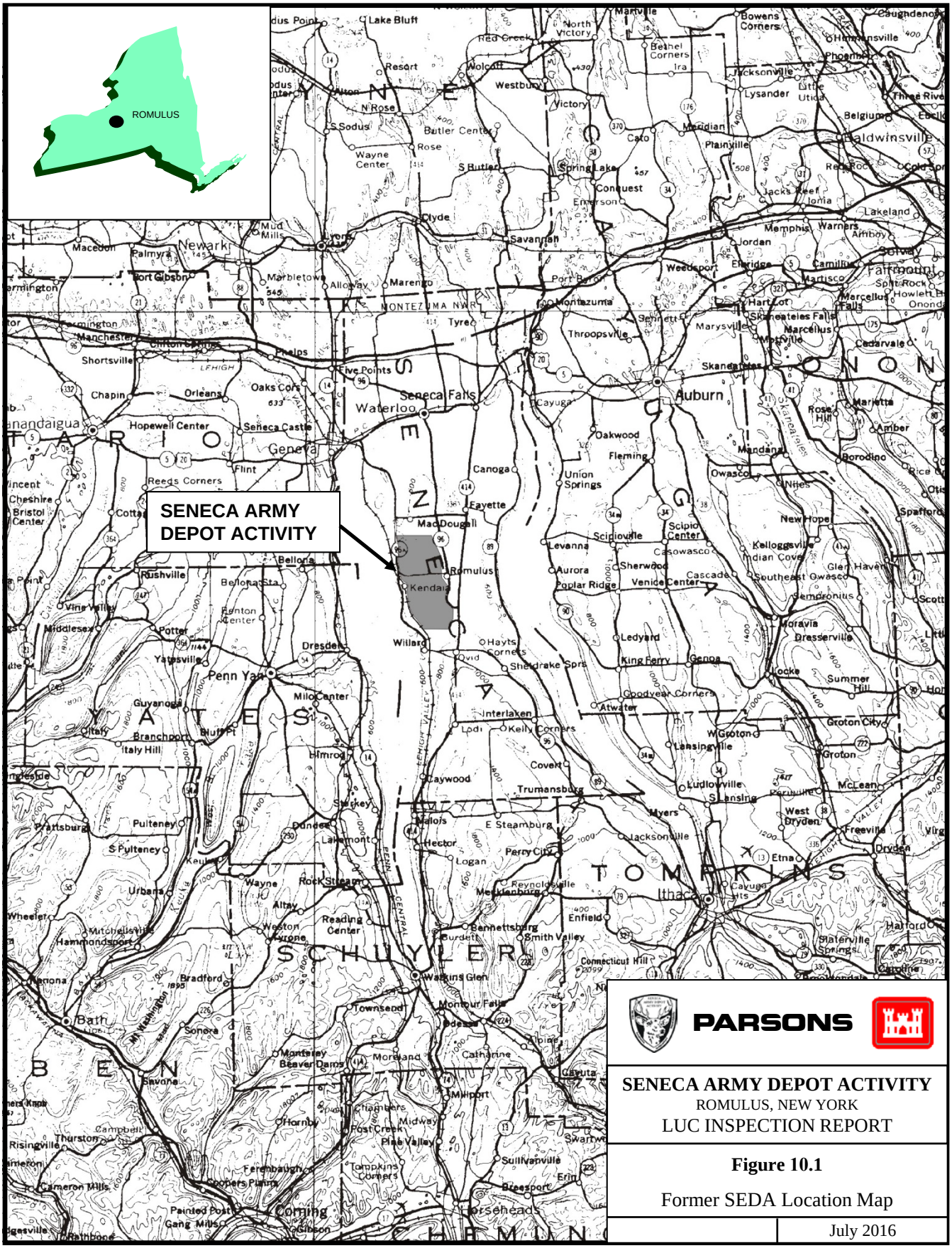
Table 10.5 - Overview of Preliminary Conceptual Site Model, SEAD-122E, Seneca Army Depot Activity

SITE DETAILS	KNOWN OR SUSPECTED CONTAMINATION SOURCE(S)	POTENTIAL/SUSPECTED LOCATION AND DISTRIBUTION	SOURCE OR EXPOSURE MEDIUM	CURRENT AND FUTURE RECEPTORS	POTENTIALLY COMPLETE EXPOSURE PATHWAY
NAME: Deicing and Aircraft Refueling Pads **Acreage**: 30 acres **Release mechanisms**: Former airfield, aircraft fueling pads **Current and Future Land Use**: Police and fire training. Future use is planned special events, industrial development and training.	PAHs and phthalates PFAS	Soil and potentially in groundwater	Soil Groundwater	Current and future onsite worker, off-site residential.	Exposure to soil Exposure to groundwater (ingestion)

Table 10.6 - Overview of Preliminary Conceptual Site Model, SEAD-26, Seneca Army Depot Activity

SITE DETAILS	KNOWN OR SUSPECTED CONTAMINATION SOURCE(S)	POTENTIAL/SUSPECTED LOCATION AND DISTRIBUTION	SOURCE OR EXPOSURE MEDIUM	CURRENT AND FUTURE RECEPTORS	POTENTIALLY COMPLETE EXPOSURE PATHWAY
NAME: SEAD-26 **Acreage**: 13 acres **Release mechanisms:** **Current and Future Land Use**: No current use. Future use is planned industrial development.	cPAHs PFAS	Potentially present in groundwater. Former soil source was removed prior to LTM.	Groundwater	Current and future onsite worker, off-site residential. Future on-site residential.	Exposure to groundwater (ingestion)

WORKSHEET #10 FIGURES



PARSONS

SENECA ARMY DEPOT ACTIVITY
ROMULUS, NEW YORK
LUC INSPECTION REPORT

Figure 10.1

Former SEDA Location Map

July 2016

P:\PIT\Projects\Huntsville Cont W912DY-08-D-0003\TO#15 - LTM and LUC\LUC Inspections\LUC Report 2016\Appendix A - Figures\Figure A1 Loc Map.pptx

N

W E

S

SEAD-23
OB Grounds

State Route 96A

SEAD-16

SEAD-25

SEAD-17

Ash Landfill OU
(SEADs 3, 6, 8,
14, & 15)

SEAD 26

Airfield and SEAD 122E

Legend

Boundary of Long Term Monitoring (LTM) Site

SEDA Boundary

2,500 1,250 0 2,500 5,000 Feet

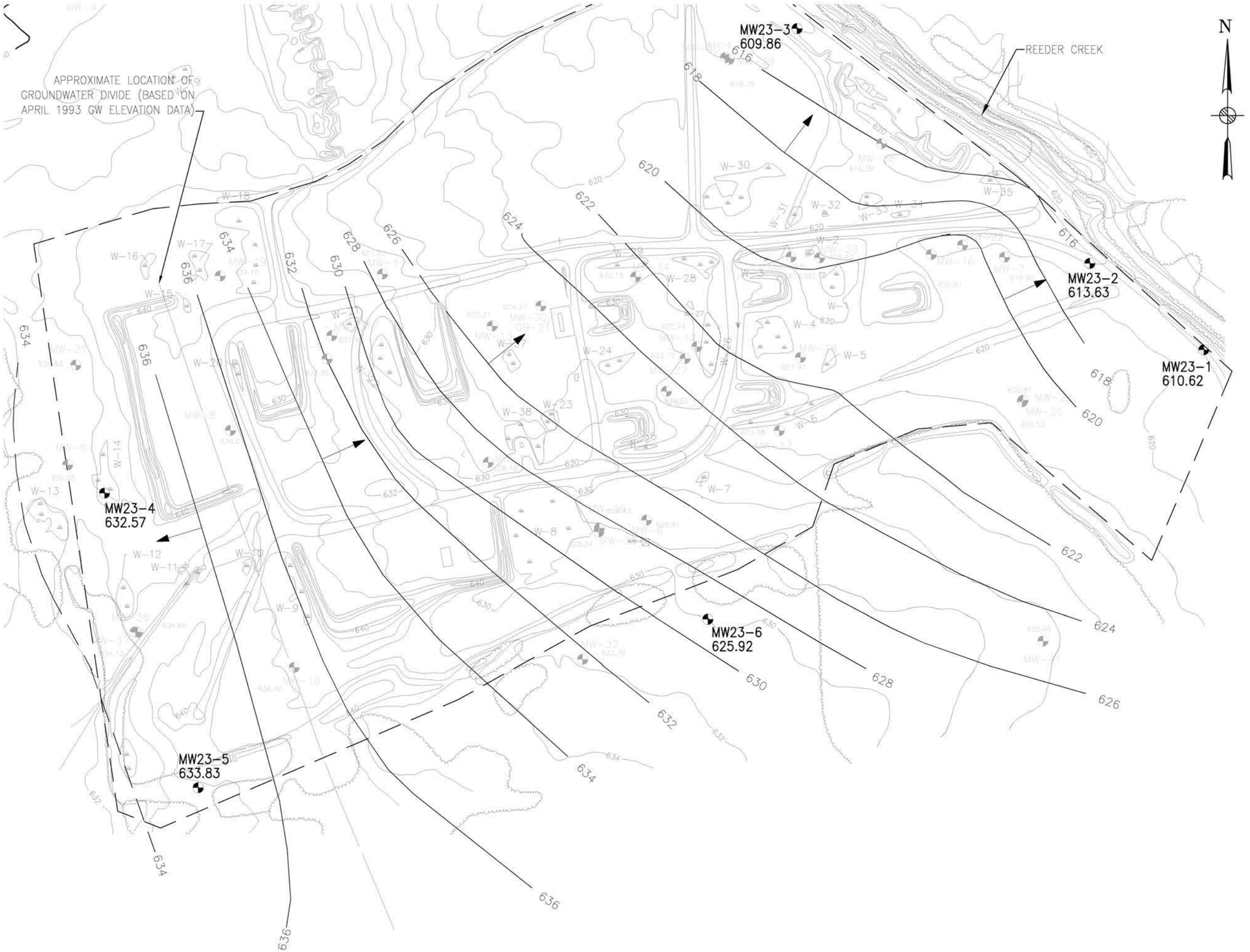
PARSONS

SENECA ARMY DEPOT ACTIVITY

FIGURE 10.2
LOCATIONS OF LTM and PFAS SITES

BBO | November 2016

Path: P:\PIT\Projects\Huntsville WERS\Seneca LTM, TO 23\01 - UFP-QAPP\! DRAFT\figures\components\SEDA LTM Sites Map.mxd



LEGEND:

- BURNING PAD DESIGNATION
- SURVEY MONUMENT
- TOPOGRAPHICAL CONTOURS
- W-1 — WETLAND & DESIGNATION
- 625.92 — CURRENT MONITORING WELL LOCATION WITH OCTOBER 2015 LTM GAUGING DATA
- HISTORICAL MONITORING WELLS WITH APRIL 1993 DATA
- 618 — HISTORIC GROUNDWATER ELEVATION CONTOUR (APRIL 1993) MSL DATUM
- GENERAL GROUNDWATER FLOW DIRECTION
- APPROXIMATE BOUNDARY AND EXTENT OF OB GROUNDS

100 0 100 200
1" = 200'

CLIENT/PROJECT TITLE

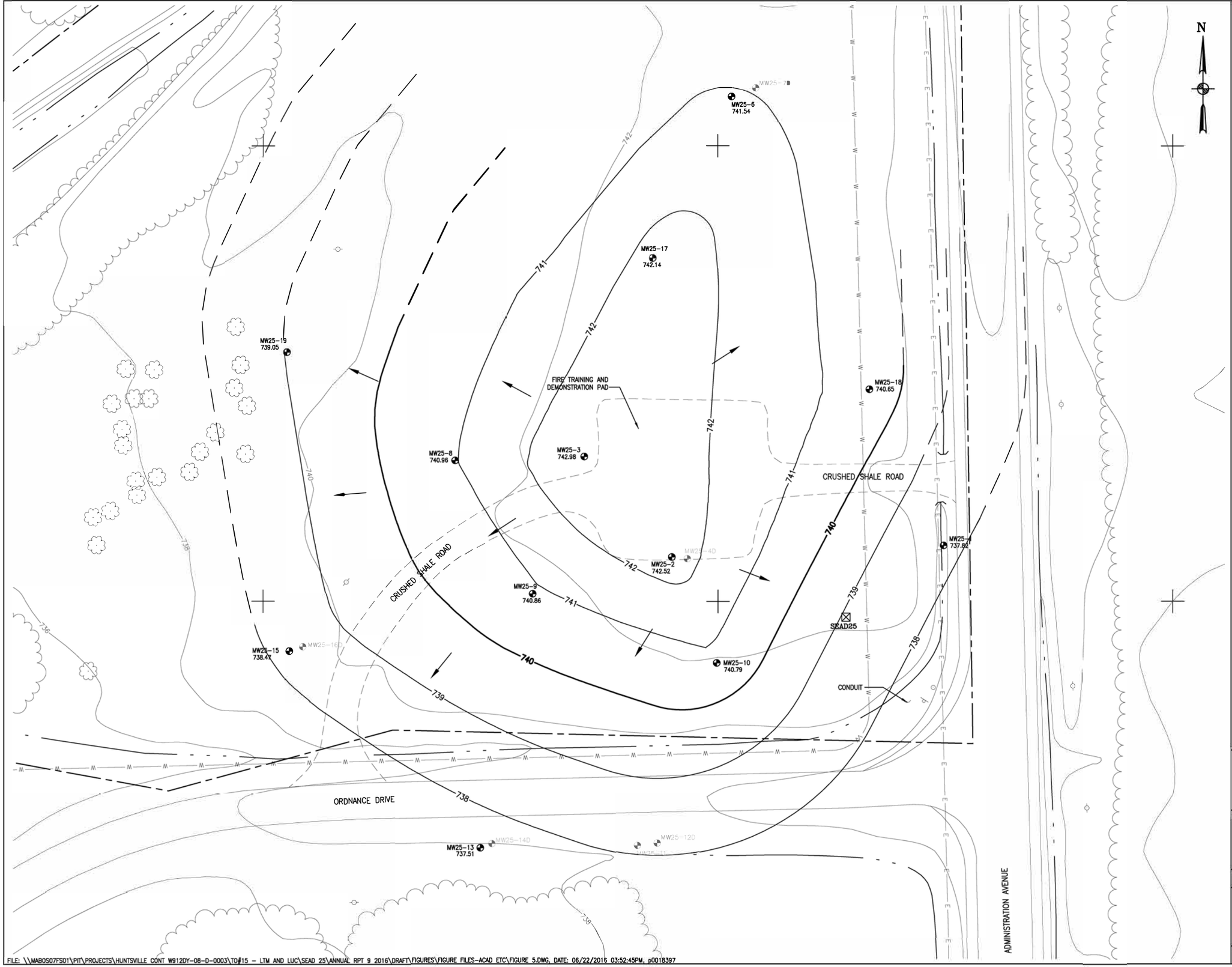
SENECA ARMY DEPOT ACTIVITY
OPEN BURNING (OB GROUNDS)
LTM 2015 ANNUAL REPORT

DEPT.	PROJECT. No.
ENVIRONMENTAL ENGINEERING	748662-01600

Figure 10.3
Historic Groundwater Contours and October 2015 Groundwater Elevations

SCALE	DATE	REV
1" = 200'	DECEMBER 2015	-

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\OB GROUNDS LTM FY 15\LTM 2015 ANNUAL REPORT\DRAFT\FIGURES\FIGU, DATE: 12/14/2015 01:47:37PM, p0018397



LEGEND

- DRAINAGE DITCH
- FENCE
- UNPAVED ROAD
- SEAD 25 BOUNDARY
- BRUSH LINE
- RAILROAD
- 760 — GROUND SURFACE ELEVATION CONTOUR
- UNDERGROUND ELECTRIC UTILITY LINE
- UNDERGROUND WATER UTILITY LINE
- ROAD SIGN
- OVERHEAD UTILITY POLE
- HYDRANT
- MANHOLE
- UTILITY BOX
- DECIDUOUS TREE
- COORD. GRID (250' GRID)
- POLE
- SEAD-25 — SURVEY MONUMENT
- MW25-19 740.1 — MONITORING WELL LOCATION & ELEVATION OF WATER TABLE
- MW25-19 — FORMER MONITORING WELL LOCATION
- 736 — GROUNDWATER CONTOUR (DASHED WHERE INFERRED)
- INDICATES PREDOMINANT FLOW DIRECTION

NOTE:

FORMER MONITORING WELLS WERE REMOVED IN SEPTEMBER 2010 AS PART OF THE WELL DECOMMISSIONING PROJECT.

25 0 25 50
(feet)

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT

ROMULUS, NEW YORK

2016 ANNUAL LONG-TERM MONITORING REPORT FOR SEAD-25

DEPT.	Dwg. No.
ENVIRONMENTAL ENGINEERING	

FIGURE 10.4

SEAD-25 GROUNDWATER CONTOURS
TILL/WEATHERED SHALE SATURATED ZONE
JUNE 2016

SCALE	DATE	REV
AS SHOWN	JUNE 2016	

FILE: \\MABOS07FS01\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\SEAD 25\ANNUAL RPT 9 2016\DRAFT\FIGURES\FIGURE FILES-ACAD ETC\FIGURE 5.DWG, DATE: 06/22/2016 03:52:45PM, p0018397

LEGEND:

- PAVED ROAD
- DIRT ROAD
- GROUND CONTOUR AND ELEVATION
- TREE
- WETLAND & DESIGNATION
- MONITORING WELL AND DESIGNATION
- RAILROAD TRACKS
- BRUSH
- CHAIN LINK FENCE
- UTILITY POLE
- APPROXIMATE LOCATION OF FIRE HYDRANT
- FUEL OR UNDERGROUND STORAGE TANK
- SURVEY MONUMENT
- ABANDONED MONITORING WELL
- APPROXIMATE LOCATION OF WATER MAIN
- PILOT STUDY BIOWALL (2005)
- SINGLE BIOWALL (2006)
- DOUBLE-WIDE BIOWALL (2006)
- ZERO VALENT IRON WALL (1998)
- GROUNDWATER CONTOUR
- EXTRAPOLATED GROUNDWATER CONTOUR
- GROUNDWATER FLOW DIRECTION

70 0 70 140
(FEET)

PARSONS

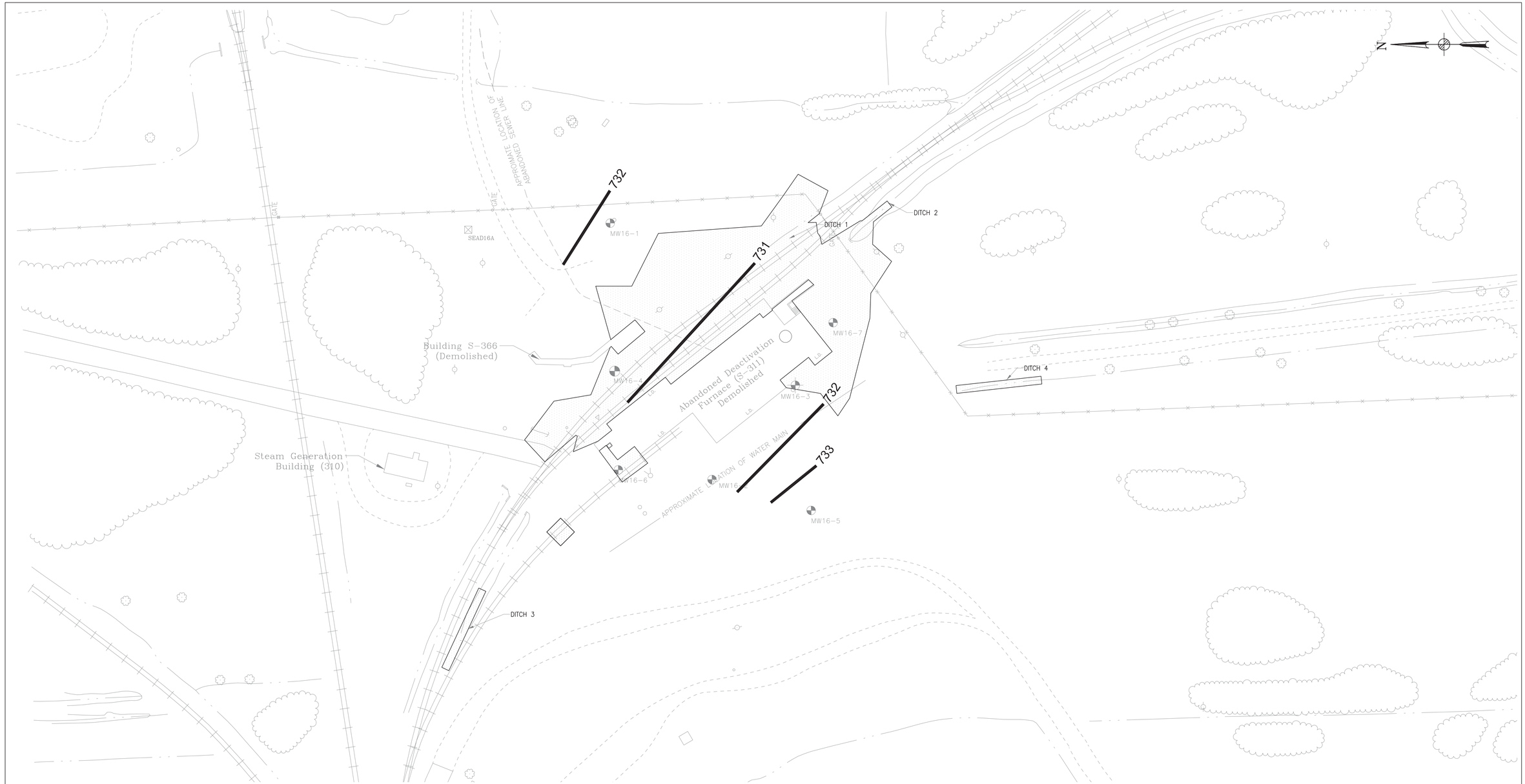
CLIENT/PROJECT TITLE
SENECA ARMY DEPOT
ASH LANDFILL
ANNUAL REPORT

DEPT. ENVIRONMENTAL ENGINEERING | Dwg. No.

FIGURE 10.5
ASH LANDFILL GROUNDWATER CONTOURS & GROUNDWATER FLOW DIRECTION DEC. 2015

SCALE | DATE MARCH 2016 | REV –

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\ASH LANDFILL LTM\YR 9 ANNUAL REPORT\DRAFT\03 - FIGURES\FIGURE 8.DWG, DATE: 08/10/2016 03:41:32PM, p0018397



LEGEND:

- MINOR WATERWAY
- MAJOR WATERWAY
- FENCE
- BRUSH LINE
- RAILROAD
- UNPAVED ROAD
- SURVEY MONUMENT
- ROAD SIGN
- DECIDOUS TREE
- L.D. LOADING DOCK
- FIRE HYDRANT
- MANHOLE
- GUIDE POST
- POLE
- UTILITY BOX
- OVERHEAD UTILITY POLE
- MAILBOX/RR SIGNAL
- MW16-5 MONITORING WELL LOCATION
- LIMITS OF EXCAVATION
- MW16-3 DESTROYED MONITORING WELL LOCATION
- 729 GROUNDWATER CONTOUR (DASHED WHERE INFERRED)

NOTE:

MONITORING WELL MW16-3 WAS DESTROYED DURING THE REMEDIAL ACTION.

50 0 50 100

SCALE IN FEET

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT ACTIVITY

ANNUAL REPORT - YEAR 8

SEAD-16 AND SEAD-17

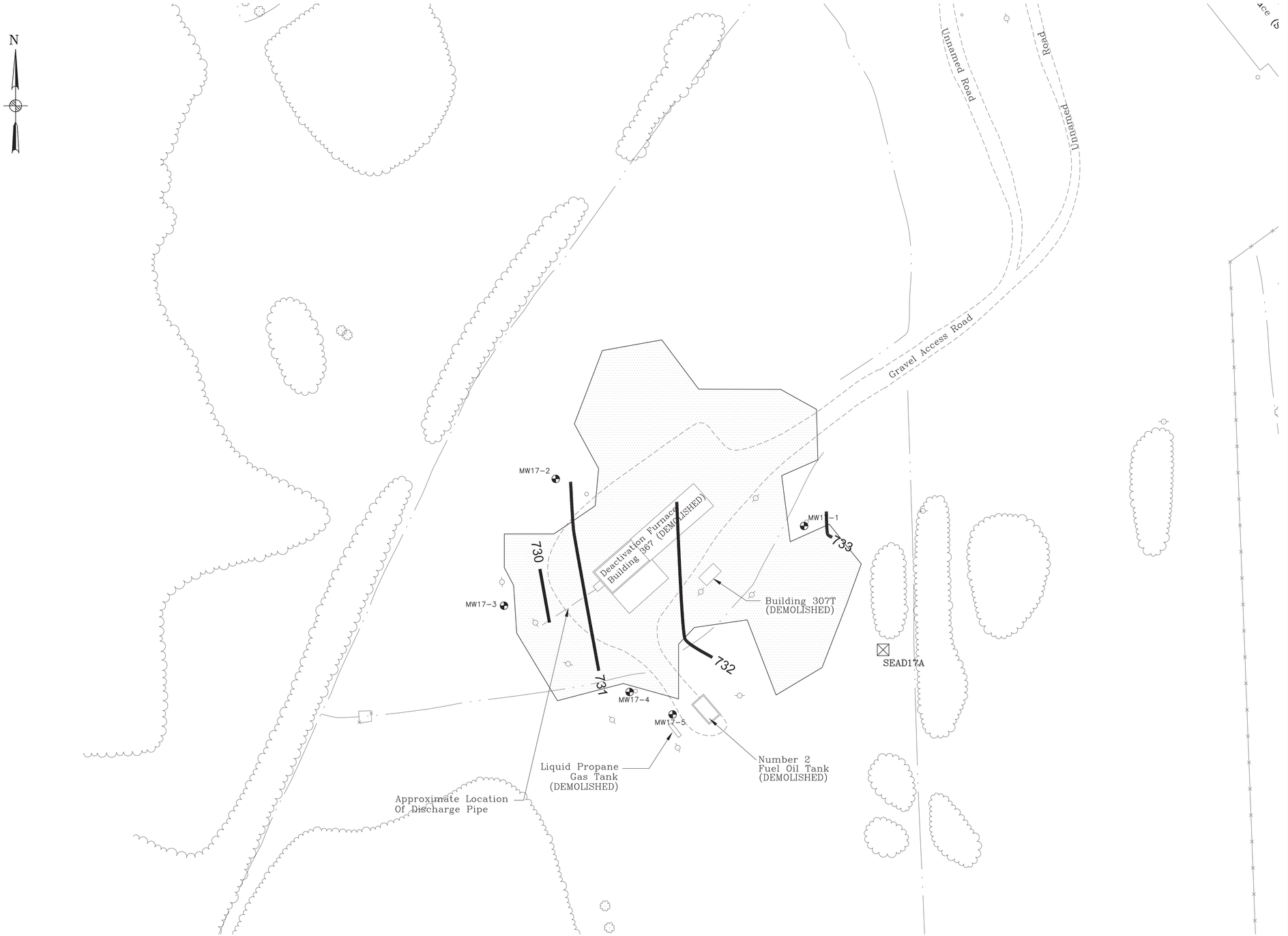
DEPT.	Dwg. No.
ENVIRONMENTAL ENGINEERING	748662-04500

FIGURE 10.6

SEAD-16 SITE PLAN

SCALE	DATE	REV
1" = 100'	FEBRUARY 2016	-

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\SEAD 16 AND 17 LTM\ANNUAL REPORT - YR 8 DEC 2015\DRAFT\FIGURES\FIGURE 3.DWG, DATE: 12/02/2016 01:16:20PM, p0018397



LEGEND:

- MINOR WATERWAY
- MAJOR WATERWAY
- FENCE
- UNPAVED ROAD
- BRUSH LINE
- RAILROAD
- 729 — GROUNDWATER CONTOUR (DASHED WHERE INFERRED)
- SURVEY MONUMENT
- ROAD SIGN
- DECIDUOUS TREE
- GUIDE POST
- FIRE HYDRANT
- MANHOLE
- MAILBOX/RR SIGNAL
- POLE
- UTILITY BOX
- OVERHEAD UTILITY POLE
- MW17-5 MONITORING WELL LOCATION
- LIMITS OF EXCAVATION

50 0 50 100
SCALE IN FEET

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT ACTIVITY
ANNUAL REPORT – YEAR 8
SEAD-16 AND SEAD-17

DEPT.	Dwg. No.
ENVIRONMENTAL ENGINEERING	748662-04500

FIGURE 10.7
SEAD-17
SITE PLAN

SCALE	DATE	REV
1" = 100'	FEBRUARY 2016	–

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 – LTM AND LUC\SEAD 16 AND 17 LTM\ANNUAL REPORT – YR 8 DEC 2015\DRAFT\FIGURES\FIGURE 4.DWG, DATE: 12/02/2016 01:17:54PM, p0018397

N
W E
S

INSTITUTIONAL AREA

DATA STORAGE / TELECOMMUNICATIONS

OB Grounds

CONSERVATION / RECREATION

PLANNED INDUSTRIAL / OFFICE DEVELOPMENT AND WAREHOUSING (PID) AREA

SEAD-16
Abandoned Deactivation Furnace

SEAD-25
Fire Training and Demonstration Pad

SEAD-17
Active Deactivation Furnace

Ash Landfill Operable Unit

FARMING

FORMER AIRFIELD COUNTY FIRE TRAINING & FACTORY

FED TO FED TRANSFER

PRISON AREA

PARSONS

SENECA ARMY DEPOT ACTIVITY

UFP-QAPP for LTM Sites

FIGURE 10.8
Future Land Use Map

September 2016

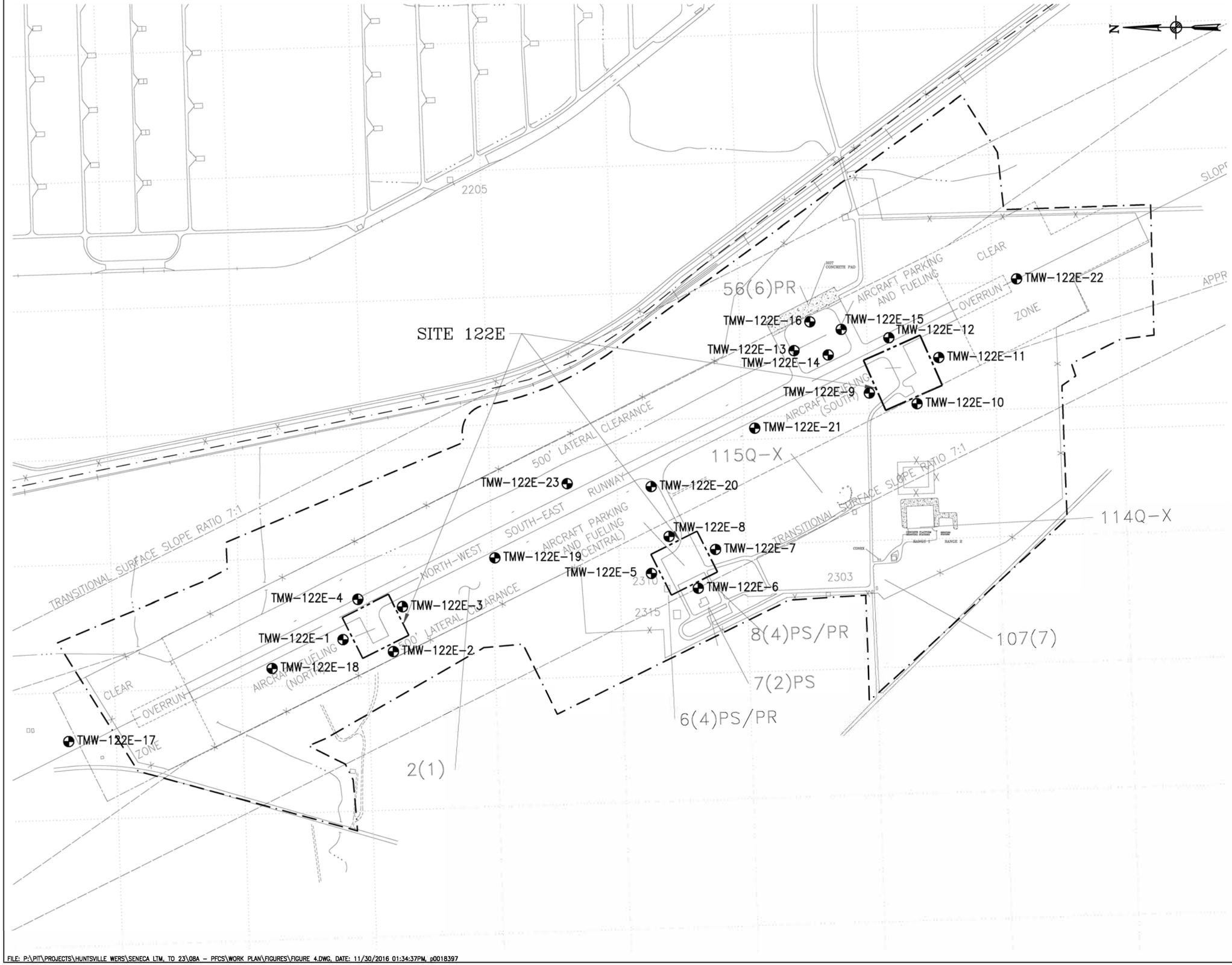
Legend

SEDA LTM Sites

SEDA Boundary

0 2,500 5,000 10,000
Feet

Path: P:\PITProjects\Huntsville WERS\Seneca LTM, TO 23\01 - UFP-QAPP\! DRAFT\figures\components\SEDA Future Land Use Map 092916.mxd



LEGEND

- DRAINAGE DITCH
- FENCE
- UNPAVED ROAD
- BRUSH LINE
- RAILROAD
- 760 — GROUND SURFACE ELEVATION CONTOUR
- UNDERGROUND ELECTRIC UTILITY LINE
- UNDERGROUND WATER UTILITY LINE
- SITE BOUNDARY
- ROAD SIGN
- OVERHEAD UTILITY POLE
- HYDRANT
- MANHOLE
- UTILITY BOX
- DECIDOUS TREE
- COORD. GRID (250' GRID)
- POLE
- PROPOSED DIRECT PUSH POINT/TEMPORARY MONITORING WELL

375 0 375 750
(feet)

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT
ROMULUS, NEW YORK
PFAS GROUNDWATER INVESTIGATION WORK PLAN

DEPT. ENVIRONMENTAL ENGINEERING | Dwg. No.

FIGURE 10.9
SEAD-122E TEMPORARY MONITORING WELL LOCATIONS

SCALE AS SHOWN | DATE NOVEMBER 2016 | REV –

FILE: P:\PIT\PROJECTS\HUNTSVILLE WERS\SENECA LTM, TO 23\08A – PFCS\WORK PLAN\FIGURES\FIGURE 4.DWG, DATE: 11/30/2016 01:34:37PM, p0018397

LEGEND

- DRAINAGE DITCH
- FENCE
- UNPAVED ROAD
- BRUSH LINE
- RAILROAD
- GROUND SURFACE ELEVATION CONTOUR
- UNDERGROUND ELECTRIC UTILITY LINE
- UNDERGROUND WATER UTILITY LINE
- SITE BOUNDARY
- ROAD SIGN
- OVERHEAD UTILITY POLE
- HYDRANT
- MANHOLE
- UTILITY BOX
- DECIDUOUS TREE
- COORD. GRID (250' GRID)
- POLE
- MW26-5 FORMER MONITORING WELL
- PROPOSED DIRECT PUSH POINT/TEMPORARY MONITORING WELL

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT

ROMULUS, NEW YORK

PFAS GROUNDWATER INVESTIGATION WORK PLAN

DEPT.	Dwg. No.
ENVIRONMENTAL ENGINEERING	

FIGURE 10.10

SEAD-26 TEMPORARY MONITORING WELL LOCATIONS

SCALE	DATE	REV
AS SHOWN	NOVEMBER 2016	—

FILE: P:\PIT\PROJECTS\HUNTSVILLE WERS\SENECA LTM, TO 23\08A - PFCS\WORK PLAN\DRAFT\FIGURES\FIGURE 3.DWG, DATE: 12/07/2016 02:56:04PM, p0018397

Worksheet #11: Data Quality Objectives

(EPA UFP-QAPP Guidance Manual, Section 2.6.1; EPA Guidance QA/G-5, Section 2.1.7)

DQOs are qualitative and quantitative statements that specify the quality and level of data required to support the decision-making processes for a project. Guidance for DQO development is contained in *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA QA/G-4), February 2006, EPA/240/B-06/001.

The overall goal of the LTM at the OB Grounds, SEAD-25, Ash Landfill, and SEAD 16/17 is to confirm there are no exceedances of groundwater cleanup standards for select COCs at each site. The purpose of the PFAS groundwater investigation is to determine the presence or absence of PFAS in groundwater as a result of firefighting training activities at the three identified SEADs. Specific DQOs for each site are outlined in **Table 11.1**. These DQOs follow the USEPA's seven-step, iterative process for DQO development. Based on the overall goal, the general project DQOs are to obtain data to sufficiently characterize the groundwater concentrations at each LTM site.

In addition to these DQOs all data collected during this project are required to attain the measurement performance criteria (MPCs) described on **Worksheet #12** to be considered adequate to support environmental decisions, unless sufficient alternative justification is provided to and accepted by the project team. Before final environmental decisions are made, data will be verified and validated as described in **Worksheets #34** through **#37**.

Table 11.1 - Data Quality Objectives and Technical Approach Summary for LTM at Seneca Army Depot Activity

SITE	STATE THE PROBLEM	IDENTIFY THE GOAL OF THE STUDY	IDENTIFY INFORMATION INPUTS	DEFINE THE BOUNDARIES OF THE STUDY	DEVELOP THE ANALYTIC APPROACH	SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA (SEE WORKSHEET #17)	DEVELOP THE DETAILED PLAN FOR OBTAINING DATA (SEE WORKSHEET #17)
OB Grounds	• Contaminated soil interred under vegetated caps throughout the site • Potential leaching of copper and lead from interred soil • Potential of soil erosion from OB Grounds into Reeder Creek	• Monitor groundwater concentrations to monitor the effectiveness of the remedial actions completed at the site with respect to preventing future groundwater quality deterioration • Inspect vegetative caps to ensure integrity and protectiveness as part of LUC inspections • Inspect Reeder Creek for accumulation of eroded sediment	• Existing groundwater data • Analytical groundwater data (Cu and Pb) and geochemical parameters • Visual inspections of vegetative caps • Visual inspections of creek bottom and banks	• The investigation will be at OB Grounds, at 6 existing monitoring wells (**Figure 10.3**). • Data will be collected during the fall months when conditions are favorable to fieldwork; site access will be coordinated with SEDA. • LTM will continue until through the fall 2016 sampling event. If COC concentrations continue to be below applicable groundwater standards then LTM will be concluded. If COCs exceed groundwater standards further LTM will be conducted.	• Review groundwater concentrations for site COCs (Worksheet #17). If there is no evidence that groundwater quality is deteriorating, then recommend future termination of LTM. LUCs and vegetative cap monitoring would continue. • If the LUC inspection team notes the presence of excessive erosion/disturbances to the vegetative cap, then the cap will be recommended for repairs • If the LUC inspection team notes evidence of soil transport from the OB Grounds to Reeder Creek or excessive accumulation of sediment in the creek bed, then a corrective action will be recommended	• NYS Class GA standards • No evidence of disturbance to vegetative cap • No evidence of soil transport from OB Grounds to Reeder Creek or accumulation of sediment in the creek bed • Prohibiting use of the land at the AOCs for residential purposes and access to and use of groundwater until applicable cleanup standards are met	• Collect groundwater samples and geochemical parameters using low-flow techniques • Visual inspections of vegetative caps and Reeder Creek
SEAD-25	• Groundwater concentrations above NYS Class GA standards	• Monitor geochemical parameters to determine the effectiveness of natural attenuation • Monitor groundwater concentrations until standards are achieved • Implementation, maintenance, inspection, and periodic reporting of LUCs	• Existing groundwater data • Analytical groundwater data (VOCs) and geochemical parameters • Visual inspection of SEAD-25	• The investigation will be at SEAD-25, at 5 existing monitoring wells (**Figure 10.4**). • Data will be collected during the spring months when conditions are favorable to fieldwork and to be consistent with timing of previous events; site access will be coordinated with SEDA. • LTM will continue until COC concentrations are at or below applicable groundwater standards for multiple rounds.	• Review groundwater concentrations for site COCs (Worksheet #17). If COC concentrations are at or below NYS Class GA standards for more than one round, then recommend future termination of LTM. LUCs would continue to be monitored. • Geochemical parameters will be compared to EPA benchmark guidance for effective natural attenuation (EPA, 1998). If site geochemical parameters are outside guidance values, a corrective action will be recommended. • The LUC inspection team will check the site to make sure LUCs are in compliance.	• Once NYS Class GA groundwater cleanup standards are achieved, the groundwater use restrictions may be eliminated upon approval of the EPA and NYSDEC • Confirm land use is not for residential purposes and there is no access to and use of groundwater until applicable cleanup standards are met	• Collect groundwater samples and geochemical parameters using low-flow techniques
Ash Landfill	• Groundwater concentrations above NYS Class GA standards • Contaminated soil interred under vegetated caps throughout the site	• Prevent exposure to off-site receptors through possible off-site migration of VOC plume • Document effectiveness of biowalls to remediate and attenuate chlorinated ethene plume • Confirm that groundwater concentrations throughout plume are decreasing to eventually meet NYSDEC Class GA groundwater standards • Monitor the integrity of vegetative cover and LUC performance objectives	• Existing groundwater data • Analytical groundwater data (VOCs) and geochemical parameters • Visual inspections of vegetative caps	• The investigation will be at Ash Landfill, at 14 existing monitoring wells (**Figure 10.5**). • Data will be collected during the months of June and December when conditions are favorable to fieldwork and to be consistent with timing of previous events; site access will be coordinated with SEDA. • LTM will continue until COC concentrations are below applicable groundwater standards for multiple rounds.	• Review groundwater concentrations for site COCs (Worksheet #17) and perform: o Long-term plume performance monitoring using existing monitoring wells PT-18, MWT-22, PT-22, PT-17, MWT-7, and PT-24; o Biowall process monitoring using existing monitoring wells; MWT-26, MWT-27, MWT-28, MWT-29, and MWT-23; and, o Confirm no exceedances of groundwater standards for COCs at the off-site compliance monitoring well (MW-56) • Review groundwater geochemical conditions to: o Monitor the long-term performance and sustainability of the biowalls o Monitor substrate depletion and geochemical conditions under which the effectiveness of the biowalls may decline o Compare with EPA benchmark guidance for effective natural attenuation (EPA, 1998). If site geochemical parameters are outside guidance values, a corrective action will be recommended. • If several lines of evidence (geochemical parameters) suggest the biowalls are no longer effective, a corrective action (e.g., biowalls recharge) will be recommended.	• NYS Class GA standards • Monitoring of both the on-site plume performance wells and off-site sentinel well will stop when GA standards for the COCs are achieved during two successive rounds of sampling the onsite plume wells. • Biowall recharge based on COC concentrations in the wall and geochemical parameters • No evidence of disturbance to vegetative cap	• Collect groundwater samples and geochemical parameters using low-flow techniques • Visual inspections of vegetative caps
SEAD-16/17	• Groundwater concentrations above NYS Class GA standards	• Monitor groundwater concentrations until standards are achieved • Implementation, maintenance, inspection, and periodic reporting of LUCs	• Existing groundwater data • Analytical groundwater data (TAL metals) and geochemical parameters	• The investigation will be at SEAD-16 at 6 existing wells and at, adjacent, SEAD-17, at 5 existing monitoring wells	• Review groundwater concentrations for site COCs (Worksheet #17). If COC concentrations are at or below NYS Class GA standards for more than one round, then recommend future termination of LTM. LUCs would continue to be monitored.	• Once NYS Class GA groundwater cleanup standards are achieved, the groundwater use restrictions may be eliminated upon approval of the EPA and NYSDEC	• Collect groundwater samples and geochemical parameters using low-flow techniques

SITE	STATE THE PROBLEM	IDENTIFY THE GOAL OF THE STUDY	IDENTIFY INFORMATION INPUTS	DEFINE THE BOUNDARIES OF THE STUDY	DEVELOP THE ANALYTIC APPROACH	SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA (SEE WORKSHEET #17)	DEVELOP THE DETAILED PLAN FOR OBTAINING DATA (SEE WORKSHEET #17)
			• Visual inspection of SEADs 16/17	(**Figure 10.6** and **Figure 10.7**). • Data will be collected during December when conditions are favorable to fieldwork and to be consistent with timing of previous events; site access will be coordinated with SEDA • The sampling frequency was recently agreed upon to change to sample next in 2019. If COCs continue to be under applicable groundwater standards LTM will be recommended to end; if COCs exceed groundwater standards, annual LTM will commence again.	• The LUC inspection team will check the site to make sure LUCs are in compliance.	• Confirm land use is not for residential purposes and there is no access to and use of groundwater until applicable cleanup standards are met	

Table 11.2 - Data Quality Objectives and Technical Approach Summary for PFAS Sampling at Seneca Army Depot Activity

SITE	STATE THE PROBLEM	IDENTIFY THE GOAL OF THE STUDY	IDENTIFY INFORMATION INPUTS	DEFINE THE BOUNDARIES OF THE STUDY	DEVELOP THE ANALYTIC APPROACH	SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA (SEE WORKSHEET #17)	DEVELOP THE DETAILED PLAN FOR OBTAINING DATA (SEE WORKSHEET #17)
SEAD-122E	• PFAS are an emerging contaminant and have a potential impact on human health and the environment	• Determine the presence or absence of PFAS in groundwater as a result of firefighting training activities	• Analytical groundwater data (PFAS)	• The investigation will be at four refueling pads and the perimeter of the airfield. • This is a site investigation to determine if PFAS are present. If PFAS concentrations exceed the acceptance criteria, further action may be proposed.	• Review groundwater concentrations for PFAS COCs (Worksheet #17). Evaluation of potential contamination and recommendations for future actions.	• EPA temporary provisional health advisory level (See Table 15.9)	• Collect groundwater grab samples via direct push and temporary wells
SEAD-25	• PFAS are an emerging contaminant and have a potential impact on human health and the environment	• Determine the presence or absence of PFAS in groundwater as a result of firefighting training activities	• Analytical groundwater data (PFAS)	• The investigation will be at 12 existing monitoring wells. • This is a site investigation to determine if PFAS are present. If PFAS concentrations exceed the acceptance criteria, further action may be proposed.	• Review groundwater concentrations for PFAS COCs (Worksheet #17). Evaluation of potential contamination and recommendations for future actions.	• EPA temporary provisional health advisory level (See Table 15.9)	• Collect groundwater samples using low-flow techniques
SEAD-26	• PFAS are an emerging contaminant and have a potential impact on human health and the environment	• Determine the presence or absence of PFAS in groundwater as a result of firefighting training activities	• Analytical groundwater data (PFAS)	• The investigation will be around the perimeter of the former fire training area. • This is a site investigation to determine if PFAS are present. If PFAS concentrations exceed the acceptance criteria, further action may be proposed.	• Review groundwater concentrations for PFAS COCs (Worksheet #17). Evaluation of potential contamination and recommendations for future actions.	• EPA temporary provisional health advisory level (See Table 15.9)	• Collect groundwater grab samples via direct push and temporary wells

Worksheet #12: Measurement Performance Criteria

(EPA UFP-QAPP Guidance Manual, Section 2.6.2; EPA Guidance QA/G-5, Section 2.1.7)

The tables below summarize the MPCs that have been established for the groundwater sampling tasks to be conducted during the LTM under this TO. The quality of the sampling procedures and laboratory results will be evaluated for compliance with DQOs through a review in accordance with the procedures described in **Worksheet #37**. The results will be summarized in a Data Usability Report (DUR). Sample collection procedures and analytical methods/SOPs are summarized on **Worksheet #21** and **Worksheet #23**, respectively.

12.1 MEASUREMENT PERFORMANCE CRITERIA FOR VOCS IN GROUNDWATER

Laboratory: Katahdin
Matrix: Groundwater
Analytical Group or Method: VOA/8260C
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	Relative Percent Difference (RPD) ≤ 30% when VOCs are detected in both samples with concentrations are ≥ sample specific Limit of Quantitation (LOQ). If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 20%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Within DoD Quality Systems Manual (QSM) Version 5.0 Appendix C Table 24 limits
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Within DoD QSM Version 5.0 Appendix C Table 24 limits
Overall accuracy/bias (contamination)	Equipment Blanks/Trip Blanks	No target analyte concentrations ≥ 1/2 LOQ
Sensitivity	LOQ verification sample (spiked at LOQ)	Recovery within ±25% of LOQ
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

12.2 MEASUREMENT PERFORMANCE CRITERIA FOR MEE IN GROUNDWATER

Laboratory: Katahdin
Matrix: Groundwater
Analytical Group or Method: MEE by RSK175
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	RPD ≤ 30% when MEE are detected in both samples with concentrations are ≥ sample specific LOQ). If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤20%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Within DoD QSM Version 5.0 Appendix C Table 42 limits
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Within DoD QSM Version 5.0 Appendix C Table 42 limits
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ 1/2 LOQ
Sensitivity	LOQ verification sample (spiked at LOQ)	Recovery within ±25% of LOQ
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

12.3 MEASUREMENT PERFORMANCE CRITERIA FOR NITRATE AND NITRITE IN GROUNDWATER

Laboratory: Katahdin
Matrix: Groundwater
Analytical Group or Method: Nitrate and Nitrite/ EPA 353.2
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	RPD ≤ 30% when the target analytes are detected in both samples ≥ sample-specific LOQ. If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 20%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	90-110 %R
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	90-110 %R
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ 1/2 LOQ
Sensitivity	LOQ verification sample (spiked at LOQ)	67-133 %R
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

12.4 MEASUREMENT PERFORMANCE CRITERIA FOR CHLORIDE AND SULFATE IN GROUNDWATER

Laboratory: Katahdin
Matrix: Groundwater
Analytical Group or Method: Chloride and Sulfate/ EPA 300.0
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	RPD ≤ 30% when the target analytes are detected in both samples ≥ sample-specific LOQ. If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 20%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	90-110 %R
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	90-110 %R
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ 1/2 LOQ
Sensitivity	LOQ verification sample (spiked at LOQ)	67-133 %R
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

12.5 MEASUREMENT PERFORMANCE CRITERIA FOR IRON, SODIUM, COPPER, AND LEAD IN GROUNDWATER

Laboratory: Katahdin
Matrix: Groundwater
Analytical Group or Method: Select Metals / EPA 6010C
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	RPD ≤ 30% when the target metals are detected in both samples ≥ sample-specific LOQ. If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 20%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Within DoD QSM Version 5.0 Appendix C Table 4 limits
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Within DoD QSM Version 5.0 Appendix C Table 4 limits
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ 1/2 LOQ
Sensitivity	LOQ verification sample (spiked at LOQ)	67-133 %R
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

12.6 MEASUREMENT PERFORMANCE CRITERIA FOR TAL METALS (EXCLUDING MERCURY) IN GROUNDWATER

Laboratory: Katahdin
Matrix: Groundwater
Analytical Group or Method: TAL Metals (Excluding Hg)/ 6020A
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	RPD ≤ 30% when the target metals are detected in both samples ≥ sample-specific LOQ. If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 20%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Within DoD QSM Version 5.0 Appendix C Table 6 limits
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Within DoD QSM Version 5.0 Appendix C Table 6 limits
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ 1/2 LOQ
Sensitivity	LOQ verification sample (spiked at LOQ)	67-133 %R
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

12.7 MEASUREMENT PERFORMANCE CRITERIA FOR TOTAL ORGANIC CARBON IN GROUNDWATER

Laboratory: Katahdin
Matrix: Groundwater
Analytical Group or Method: Total Organic Carbon/ 9060A
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	RPD ≤ 30% when the analyte is detected in both samples ≥ sample-specific LOQ. If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 30%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	80-120 %R
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	75-125 %R
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ 1/2 LOQ
Sensitivity	LOQ verification sample (spiked at LOQ)	67-133 %R
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

12.8 MEASUREMENT PERFORMANCE CRITERIA FOR MERCURY IN GROUNDWATER

Laboratory: Katahdin
Matrix: Groundwater
Analytical Group or Method: Mercury/ 7470A
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	RPD ≤ 30% when mercury is detected in both samples ≥ sample-specific LOQ. If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 20%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Within DoD QSM Version 5.0 Appendix C Table 12 limits
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Within DoD QSM Version 5.0 Appendix C Table 12 limits
Overall accuracy/bias (contamination)	Equipment Blanks	No mercury concentrations ≥ 1/2 LOQ
Sensitivity	LOQ verification sample (spiked at LOQ)	67-133 %R
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

12.9 MEASUREMENT PERFORMANCE CRITERIA FOR PERFLUORINATED COMPOUNDS (PFAS) IN GROUNDWATER

Laboratory: TestAmerica- W. Sacramento
Matrix: Groundwater
Analytical Group or Method: PFAS/ EPA 537 Modified
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	RPD ≤ 30% when the analyte is detected in both samples ≥ sample-specific LOQ. If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 30%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	70-130% Recovery
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	70-130% Recovery
Overall accuracy/bias (contamination)	Equipment Blanks, Trip Blanks and Field Blanks	No target analyte concentrations ≥ 1/2 LOQ
Sensitivity	LOQ verification sample (spiked at LOQ)	Performed quarterly per the requirements included in the DoD QSM, version 5.0
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

Worksheet #13: Secondary Data Criteria and Limitations

(EPA UFP-QAPP Guidance Manual, Section 2.7)

This table lists the secondary data used to support decision making during this investigation.

SECONDARY DATA	DATA SOURCE	DATA GENERATOR(S)	HOW DATA WILL BE USED	LIMITATIONS ON DATA USE
Background metals concentrations in groundwater	Parsons Engineering Science (1995)	USACE, New York District; and Parsons.	Data will be used to provide background metals concentrations for results comparison.	None.

Worksheets #14 & 16: Project Tasks and Schedule

(EPA UFP-QAPP Guidance Manual, Section 2.8.2, EPA Guidance QA/G-5, Section 2.1.4)

The activities to be conducted at Seneca Army Depot Activity to achieve the project DQOs (**Worksheet #11**) comprise of one primary component: to obtain analytical data to monitor the effectiveness of the implemented remedies at the OB Grounds, SEAD-25, Ash Landfill, and SEAD-16/17. While this primary component is the focus of the project, the field operations involve multiple elements, or "DFWs," that will be required to achieve the project goals. This subchapter provides a summary of these DFWs and the associated component tasks. A detailed discussion of the primary project component at each site and the related DFWs is included on **Worksheet #17**, and the specific field procedures to be used for the activities described in this summary are included in the various SOPs appended to this UFP-QAPP. The project schedules will be provided in site specific planning documents.

DEFINABLE FEATURE OF WORK (ACTIVITY)	ASSOCIATED TASKS	RELATED SOPS
Mobilization	• Preparation (review plans, make travel arrangements, etc.) • Mobilize equipment and vehicles to the site • Set up site communications • Conduct site-specific training and briefing for required field personnel	--
Site Preparation	• Set up and calibrate sampling equipment • Prepare sample bottles and labels	--
Sampling and Analysis	• Collect and analyze groundwater samples • Conduct QC evaluation of analytical data for validation • Document data validation and sample results	• Parsons SOPs (Worksheet #21): • Analytical SOPs (Worksheet #23)
LUC Inspections	• Inspect soil caps at the OB Grounds and Ash Landfill • Inspect Reeder Creek at the OB Grounds • Inspect each site for LUC compliance	--
Demobilization	• Upon completion of field activities all personnel, equipment and materials will be removed from the site	--
Reports	• OB Grounds: Annual reports, typically two months after sampling event, which include groundwater analytical results, vegetative cap inspection and creek inspection results, and LTM recommendations. • SEAD-25: Annual reports, typically two months after sampling event, which include groundwater analytical results and LTM recommendations. • Ash Landfill: Semi-annual reports (one technical report and one annual report), typically two months after sampling event, which include groundwater analytical results, biowall recharge evaluation, vegetative cap inspection, and LTM recommendations. • SEAD-16/17: Annual reports, typically two months after sampling event, which include groundwater analytical results and LTM recommendations. • Annual LUC Inspection reports: Yearly status of the LUCs at each site. • SI Report: Summary of completed field activities, summary of data, including presentation on tables and figures, and evaluation of contamination and recommendations for future actions.	

Worksheet #15: Project Action Limits and Laboratory-Specific Detection / Quantitation Limits

(EPA UFP-QAPP Guidance Manual, Section 2.8.1)

This worksheet provides the parameters to be analyzed and their associated limits of quantitation (LOQ), limits of detection (LOD), and detection limits (DL) in order to satisfy the overall DQOs. The PALs, as referenced in the DQOs on **Worksheet #11**, are also included. The Project Action Limits (PALs) for this project were selected based on the lowest enforceable standard between the New York State Class GA (NYS Class GA) Ambient Water Quality Standards (NYSDEC, 1998) and the USEPA maximum contaminant level (MCL) (EPA, 2016a).

In some cases, the LOQ is greater than the screening value due to limitations in the analytical method. This is common in some analyses due to sample preparation and analytical limitations. The selected laboratory is using the appropriate analytical method and no approved alternative method has been identified that would achieve lower LOD/LOQs. This could lead to a situation where the analyte is present at a concentration greater than the screening value, but is reported as "not detected or estimated," leading to a potential underestimate of risk. In such a case, detections between the LOQ and LOD are J qualified and addressed as detects, the data will be considered usable for determining nature and extent and all detects will be used for planning purposes. If the sensitivity requirements are not met for a particular analyte, the Parsons Team will evaluate whether the data can still be used for project decisions. 1,2-DCA is the only analyte with an LOD/LOQ greater than the PAL that has been previously detected at the Ash Landfill. While 1,2-DCA has been detected at the site, is not a site-related compound. Rather it is related to the reductive dechlorination process occurring at the Ash Landfill. The compound is reported in the semi-annual reports and will be monitored for any increasing trends. No other analytes with LOD/LOQs greater than the PAL were previously identified as COCs at the Ash Landfill; therefore, the potential likelihood that the analytes are present is limited. The LOD/LOQs are considered sufficient for determining data usability at this site. Any analytes that are not detected in any well at the site will be considered to not be present at the site and used for site-related decisions.

Table 15.1 - Project Action Limits and Katahdin Reference Limits for VOCs in Groundwater (Method SW-846 8260C)

ANALYTE	PROJECT ACTION LIMIT (µG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (3) (µG/L)	LOD (µG/L)	DL (µG/L)
1,1,1-Trichloroethane	5	NYS GA	1.0	0.50	0.20
1,1,2,2-Tetrachloroethane	5	NYS GA	1.0	0.50	0.38
1,1,2-Trichloro-1,2,2-Trifluoroethane	5	NYS GA	1.0	0.50	0.31
1,1,2-Trichloroethane	1	NYS GA	1.0	0.50	0.33
1,1-Dichloroethane	5	NYS GA	1.0	0.50	0.21
1,1-Dichloroethene	5	NYS GA	1.0	0.50	0.35
1,2,4-Trichlorobenzene	5	NYS GA	1.0	0.50	0.37
1,2-Dibromo-3-chloropropane	0.04	NYS GA	1.0	0.75	0.50
1,2-Dibromoethane	0.0006	NYS GA	1.0	0.50	0.20
1,2-Dichlorobenzene	3	NYS GA	1.0	0.50	0.15
1,2-Dichloroethane	0.6	NYS GA	1.0	0.50	0.20
1,2-Dichloropropane	1	NYS GA	1.0	0.50	0.25
1,3-Dichlorobenzene	3	NYS GA	1.0	0.50	0.26
1,4-Dichlorobenzene	3	NYS GA	1.0	0.50	0.24
Acetone	50	NYS GA	5.0	2.5	2.2
Benzene	1	NYS GA	1.0	0.50	0.26

ANALYTE	PROJECT ACTION LIMIT (µG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (3) (µG/L)	LOD (µG/L)	DL (µG/L)
Bromodichloromethane	80	MCL	1.0	0.50	0.33
Bromoform	80	MCL	1.0	0.50	0.23
Carbon disulfide	NA	NA	1.0	0.50	0.25
Carbon tetrachloride	5	NYS GA	1.0	0.50	0.22
Chlorobenzene	5	NYS GA	1.0	0.50	0.22
Chlorodibromomethane	80	MCL	1.0	0.50	0.30
Chloroethane	5	NYS GA	2.0	1.0	0.55
Chloroform	7	NYS GA	1.0	0.50	0.32
Cis-1,2-Dichloroethene	5	NYS GA	1.0	0.50	0.21
Cis-1,3-Dichloropropene	0.4	NYS GA	1.0	0.50	0.19
Cyclohexane	NA	NA	1.0	0.50	0.31
Dichlorodifluoromethane	5	NYS GA	2.0	1.0	0.24
Ethyl benzene	5	NYS GA	1.0	0.50	0.21
Isopropylbenzene	5	NYS GA	1.0	0.50	0.23
Methyl Acetate	NA	NA	1.0	0.75	0.53
Methyl bromide	5	NYS GA	2.0	1.0	0.49
Methyl butyl ketone	50	NYS GA	5.0	2.5	1.7
Methyl chloride	5	NYS GA	2.0	1.0	0.36
Methyl cyclohexane	NA	NA	1.0	0.50	0.30
Methyl ethyl ketone	50	NYS GA	5.0	2.5	1.3
Methyl isobutyl ketone	NA	NA	5.0	2.5	1.3
Methyl Tertbutyl Ether	NA	NA	1.0	0.50	0.36
Methylene chloride	5	NYS GA	5.0	2.5	1.1
Styrene	5	NYS GA	1.0	0.50	0.23
Tetrachloroethene	5	NYS GA	1.0	0.50	0.40
Toluene	5	NYS GA	1.0	0.50	0.27
Total Xylenes	5	NYS GA	3.0	1.5	0.25
Trans-1,2-Dichloroethene	5	NYS GA	1.0	0.50	0.25
Trans-1,3-Dichloropropene	0.4	NYS GA	1.0	0.50	0.20
Trichloroethene	5	NYS GA	1.0	0.50	0.28
Trichlorofluoromethane	5	NYS GA	2.0	1.0	0.24
Vinyl chloride	2	NYS GA	2.0	1.0	0.25

(1) PAL was determined by selecting the lower value between NYS Class GA and EPA MCL standards.
https://www.epa.gov/ground-water-and-drinking-water/table-regulated-drinking-water-contaminants
http://www.dec.ny.gov/docs/water_pdf/togs111.pdf

(2) Gray highlighted values indicate that the value is greater than the PAL.

NA = Not available

Table 15.2 - Project Action Limits and Katahdin Reference Limits for MEE in Groundwater (Method RSK 175)

ANALYTE	PROJECT ACTION LIMIT (µG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (µG/L)	LOD (µG/L)	DL (µG/L)
Methane	NA	NA	10	5.0	0.68
Ethylene	NA	NA	10	5.0	0.69
Ethane	NA	NA	10	5.0	0.58

(1) PAL was determined by selecting the lower value between NYS Class GA and EPA MCL standards
(2) NA= Not available.
https://www.epa.gov/ground-water-and-drinking-water/table-regulated-drinking-water-contaminants
http://www.dec.ny.gov/docs/water_pdf/togs111.pdf

Table 15.3 - Project Action Limits and Katahdin Reference Limits for Nitrate and Nitrite in Groundwater (EPA Method 353.2)

ANALYTE	PROJECT ACTION LIMIT (MG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (2) (MG/L)	LOD (MG/L)	DL (MG/L)
Nitrate	10	MCL	0.050	0.025	0.015
Nitrite	1	MCL	0.050	0.025	0.0032

(1) PAL was determined by selecting the lower value between NYS Class GA and EPA MCL standards.
https://www.epa.gov/ground-water-and-drinking-water/table-regulated-drinking-water-contaminants
http://www.dec.ny.gov/docs/water_pdf/togs111.pdf

Table 15.4 - Project Action Limits and Katahdin Reference Limits for Chloride and Sulfate in Groundwater (EPA Method 300.0)

ANALYTE	PROJECT ACTION LIMIT (MG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (MG/L)	LOD (MG/L)	DL (MG/L)
Chloride	250	NYS GA	2.0	1.0	0.099
Sulfate	250	NYS GA	1.0	0.50	0.064

(1) PAL was determined by selecting the lower value between NYS Class GA and EPA MCL standards.
https://www.epa.gov/ground-water-and-drinking-water/table-regulated-drinking-water-contaminants
http://www.dec.ny.gov/docs/water_pdf/togs111.pdf

Table 15.5 - Project Action Limits and Katahdin Reference Limits for Select Metals in Groundwater (Method SW-846 6010C)

ANALYTE	PROJECT ACTION LIMIT (µG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (µG/L)	LOD (µG/L)	DL (µG/L)
Copper	200	NYS GA	25	10	0.63
Iron	300	NYS GA	100	80	5.4
Lead	15	MCL	5.0	4.0	1.1
Sodium	20,000	NYS GA	1000	500	24

(1) PAL was determined by selecting the lower value between NYS Class GA and EPA MCL standards.
https://www.epa.gov/ground-water-and-drinking-water/table-regulated-drinking-water-contaminants
http://www.dec.ny.gov/docs/water_pdf/togs111.pdf

Table 15.6 - Project Action Limits and Katahdin Reference Limits for TAL Metals, Excluding Mercury (Method SW-846 6020A)

ANALYTE	PROJECT ACTION LIMIT (µG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (µG/L)	LOD (µG/L)	DL (µG/L)
Aluminum	NA	NA	100	40	4.4
Antimony	3	NYS GA	1.0	0.50	0.054
Arsenic	10	MCL	5.0	4.0	2.2
Barium	1,000	NYS GA	2.0	1.0	0.27
Beryllium	4	MCL	1.0	0.20	0.034
Cadmium	5	MCL	1.0	0.20	0.030
Calcium	NA	NA	100	80	20
Chromium	50	NYS GA	4.0	3.0	0.22
Cobalt	NA	NA	1.0	0.30	0.060
Copper	200	NYS GA	3.0	2.0	0.18
Iron	300	NYS GA	100	60	13
Lead	15	MCL	1.0	0.50	0.074
Magnesium	NA	NA	100	80	7.8
Manganese	300	NYS GA	2.0	1.0	0.35
Nickel	100	NYS GA	2.0	1.2	0.15
Potassium	NA	NA	1000	400	31
Selenium	10	NYS GA	5.0	3.0	0.19
Silver	50	NYS GA	1.0	0.40	0.050
Sodium	NA	NA	1000	400	18
Thallium	2	MCL	1.0	0.40	0.060
Vanadium	NA	NA	5.0	4.0	0.51
Zinc	NA	NA	10	8.0	3.9

(1) PAL was determined by selecting the lower value between NYS Class GA and EPA MCL standards.
https://www.epa.gov/ground-water-and-drinking-water/table-regulated-drinking-water-contaminants
http://www.dec.ny.gov/docs/water_pdf/togs111.pdf
NA=Not available

Table 15.7 - Project Action Limits and Katahdin Reference Limits for TOC in Groundwater (Method SW-846 9060A)

ANALYTE	PROJECT ACTION LIMIT (MG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (MG/L)	LOD (MG/L)	DL (MG/L)
TOC	NA	NA	1.0	0.50	0.10

(1) PAL was determined by selecting the lower value between NYS Class GA and EPA MCL standards.NA= Not available
https://www.epa.gov/ground-water-and-drinking-water/table-regulated-drinking-water-contaminants
http://www.dec.ny.gov/docs/water_pdf/togs111.pdf

Table 15.8 - Project Action Limits and Katahdin Reference Limits for Mercury in Groundwater (Method SW-846 7470A)

ANALYTE	PROJECT ACTION LIMIT (µG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (µG/L)	LOD (µG/L)	DL (µG/L)
Mercury	0.7	NYS GA	0.20	0.10	0.013

(1) PAL was determined by selecting the lower value between NYS Class GA and EPA MCL standards.
https://www.epa.gov/ground-water-and-drinking-water/table-regulated-drinking-water-contaminants
http://www.dec.ny.gov/docs/water_pdf/togs111.pdf

Table 15.9 - Project Action Limits and TestAmerica- W. Sacramento Reference Limits for PFAS (EPA Method 537)

ANALYTE	PROJECT ACTION LIMIT (nG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (nG/L)	LOD (nG/L)	DL (nG/L)
Perfluorohexanoic acid (PFHxA)	NA	NA	2.50	2.00	0.786
Perfluoroheptanoic acid (PFHpA)	NA	NA	2.50	2.00	0.802
Perfluorooctanoic acid (PFOA)	70	EPA Health Advisory Limit (2)	2.50	2.00	0.748
Perfluorononanoic acid (PFNA)	NA	NA	2.50	2.00	0.654
Perfluorodecanoic acid (PFDA)	NA	NA	2.50	1.00	0.440
Perfluoroundecanoic acid (PFUnA)	NA	NA	2.50	2.00	0.748
Perfluorododecanoic acid (PFDoA)	NA	NA	2.50	2.00	0.584
Perfluorotridecanoic Acid (PFTriA)	NA	NA	2.50	2.00	0.551
Perfluorotetradecanoic acid (PFTeA)	NA	NA	2.50	1.00	0.400
Perfluorobutanesulfonic acid (PFBS)	NA	NA	2.50	2.00	0.918
Perfluorohexanesulfonic acid (PFHxS)	NA	NA	2.50	2.00	0.870
Perfluorooctanesulfonic acid (PFOS)	70	EPA Health Advisory Limit (2)	4.00	3.00	1.276
N-ethyl perfluorooctane sulfonamidoacetic acid (NEtFOSAA)	NA	NA	20.0	5.02	15.0
N-methyl perfluorooctane sulfonamidoacetic acid (NMeFOSAA)	NA	NA	20.0	5.64	15.0

1) NA – No NYSDEC Class GA or EPA MCL available.
2) EPA Health Advisory Limits are drinking water limits (EPA, 2016b).

Worksheet #17: Sampling Design and Rationale

(EPA UFP-QAPP Guidance Manual, Section 3.1.1)

17.1 INTRODUCTION

The project objectives for the LTM at Seneca Army Depot Activity are to obtain data to monitor the effectiveness of the implemented remedies at the OB Grounds, SEAD-25, Ash Landfill, and SEAD-16/17. Additional activities to be conducted separately from LTM include sampling of emerging contaminants (perfluroalkyl substances {PFAS]) at sites where Aqueous Film Forming Foams (AFFF) (e.g., firefighting foams) may have been used at Seneca: Fire Training and Demonstration Pad (SEAD-25), Fire Training Pit (SEAD-26), and Airfield and Fuel Pads (SEAD-122E). The boundaries of each Site are shown on **Figures 10.3 through 10.10**, respectively. The general technical approach is based on the CSM for each Site, which is described on **Worksheet #10**.

This worksheet describes the project design and the tasks that will be required to successfully complete field operations during this project and achieve the DQOs described on **Worksheet #11**. These DQOs include a design for obtaining groundwater data for all six Sites in addition to visual inspections at the OB Grounds and Ash Landfill. The design for obtaining data described in the last column of the DQO tables on **Worksheet #11** summarizes the technical approach for each investigation area at SEDA, including visual surveys and the collection of environmental samples. The technical approach for each Site is also summarized in Section 17.2.

The field operations involve multiple elements, or "definable features of work," that will be required to achieve the project goals. These definable features of work involved with LTM and PFAS sampling are listed on **Worksheet #14** and they are explained further in this worksheet, with references to relevant SOPs (**Worksheet #21** and **Appendix A and D**), MPCs (**Worksheet #12**), and other sections of the UFP-QAPP, as necessary.

17.2 DEFINEABLE FEATURES OF WORK

17.2.1 MOBILIZATION

Preparations for mobilization will commence upon approval of this UFP-QAPP. LTM and PFAS sampling will be conducted under separate mobilizations. Upon receipt of document approval, the field team will be notified, travel and lodging arrangements will be made, and the requisite copies of applicable documents will be assembled. The field management team will have already reviewed the available documentation relating to the site and this UFP-QAPP. Based on historic data and findings, the field teams will be mobilized to Seneca around the following timeframes as presented in **Table 17.1** below to ensure optimal sampling conditions.

Table 17.1 – Mobilization Schedule

SITE	SAMPLE COLLECTION TIMEFRAME (1)
OB Grounds	October
SEAD 25	March
Ash Landfill	June, December
SEAD 16/17	December
SEAD-122E, SEAD-25, SEAD-26	March/April

(1) Timeframes may shift if deemed appropriate or necessary

Equipment and materials will either be shipped to the site via commercial carrier, transported to the site by the field team, or obtained locally, as appropriate. Equipment may include, but is not limited to, sampling supplies, sample

containers, documents, first aid kits, fire extinguishers, digital cameras, etc. Site vehicles will be rented and, in most cases, will be four-wheel drive vehicles that will accommodate all site personnel and equipment. Drilling equipment will be brought to the site by a subcontractor.

The primary means of onsite communication will be achieved using cellular telephones. If separated from one another, each member of the field sampling team will have an operational cell phone available at all times for emergency use.

Prior to field activities, all field team members will be given site-specific training involving:

- Activities to be performed;
- Safe work practices; and
- Installation-specific procedures.

In addition to this training, the field team will be briefed each day prior to commencement of field activities by the field team lead. Daily briefings will include a discussion of weather conditions and the coming day's activities.

17.2.2 SITE PREPARATION

The field teams will utilize the field office on-site to prepare for the groundwater sample collection and inspection activities. The sampling equipment will be calibrated and inspected daily to ensure proper functionality (**Worksheet #22**). The appropriate number of sample bottles, and the respective bottle labels will also be prepared at the field office (**Worksheet #18**).

17.2.3 SAMPLING AND ANALYSIS

The LTM objective of this task is to obtain data to monitor the effectiveness of the implemented remedies at the OB Grounds, SEAD-25, Ash Landfill, and SEAD-16/17. During separate mobilizations, additional sites will be sampled for the presence or absence of PFAS. In addition to the sampling and analysis descriptions provided below for each site, the specific details are addressed in greater detail on **Worksheet #18** and in Parsons SOP ENV-02 and PFAS SOPs (**Appendix D**), and the analytical procedures are summarized on **Worksheets #19** and **#30** and **Worksheet #23**. The field sampling forms to be filled out at each monitoring well are located **Appendix B**.

OB Grounds

As described on **Worksheet #10**, LTM at the OB Grounds has been ongoing since 2007 to monitor if metals (i.e., lead and copper) are potentially present in the groundwater as a result of leaching from the interred soil. To continue performing LTM at the OB Grounds, groundwater samples will be collected from the six on-site existing monitoring wells (**Figure 10.3**) using a low-flow peristaltic pump on an annual basis. The wells will be purged until stabilization is achieved before collecting the sample in accordance with SOP ENV-02 (**Worksheet #21**). All six existing monitoring wells will be gauged to document the water level across the site.

The groundwater samples will be analyzed for total copper and total lead using analytical method USEPA SW846 Method 6010C. Additionally, during field sampling, the geochemical parameters presented in **Table 17.2** will be recorded for the duration of low-flow sampling until stabilization for each groundwater sample.

Table 17.2 – Geochemical Parameters

EQUIPMENT	PARAMETER	STABLILIZATION REQUIREMENTS
Horiba U-52 Multi-Parameter Water Quality Meter	• Oxidation reduction potential (ORP) • pH • Specific Conductivity	• ± 20 mV • ± 0.2 pH units • ± 3% of reading
YSI 85 Meter	• Dissolved oxygen (DO) • Temperature	• ± 10% of reading or ± 0.2 mg/L, whichever is greater • ± 1 °C
Lamotte 2020 Turbidity Meter (or similar)	• Turbidity	• ± 10% of prior reading or ± 1.0 NTU

SEAD 25

As described on **Worksheet #10**, LTM at SEAD 25 has been ongoing since 2006 as part of the continuing post-closure monitoring and maintenance operations. To monitor the effectiveness of natural attenuation following the source removal of impacted soil from SEAD 25, LTM will continue under this TO on an annual basis. Water level measurements will be collected from all 12 existing monitoring wells at SEAD-25; however, as described in Section 10.5.2, the groundwater samples from only five wells (**Figure 10.4**) (**Worksheet #18**) will be analyzed. A low-flow bladder pump will be used to purge the wells, to minimize disturbances and limit turbidity. The monitoring wells will be purged and samples will be collected in accordance with SOP ENV-02 (**Worksheet #21**).

The analytes and the respective analysis to be used at SEAD-25 are summarized below:

- VOCs – EPA SW846 Method 8260C
- MEE – RSK-175
- Nitrate and Nitrite – EPA Method 353.2
- Chloride – EPA Method 300.0
- Sulfate – EPA Method 300.0
- Iron – EPA SW846 Method 6010C
- Sodium – EPA SW846 Method 6010C

In addition to the geochemical parameters presented in **Table 17.2**, sulfide concentrations will be measured in the field using a Hach® colorimeter test at the well locations. Geochemical parameters, MEE, nitrate/nitrite, chloride, sulfate, sulfide, and iron concentrations will be used to evaluate the effectiveness of natural attenuation at SEAD-25. A comprehensive list showing analyses to be performed at each well is included on **Worksheet #18**.

Ash Landfill

As described on **Worksheet #10**, LTM at the Ash Landfill has been ongoing since 2007. The three types of LTM being performed at the Ash Landfill are: 1) plume performance monitoring, 2) biowall process monitoring, and 3) off-site compliance monitoring. On-site performance monitoring is being conducted to measure groundwater contaminant concentrations and to evaluate the effectiveness of the biowall remedy for the Ash Landfill. The objectives of performance and compliance monitoring are as follows:

- Confirm that there are no exceedances of groundwater standards for COCs at the off-site compliance monitoring well MW-56 (**Figure 10.5**);
- Document the effectiveness of the biowalls to remediate and attenuate the chlorinated ethene plume (biowall performance monitoring wells MWT-26, MWT-27, MWT-28, MWT-29, MWT-23); and
- Confirm that groundwater concentrations throughout the plume are decreasing to eventually meet NYSDEC Class GA groundwater standards (plume performance walls PT-18A, MWT-25, MWT-28, MWT-29, MWT-22, PT-22, MWT-23, MWT-24, PT-17, MWT-7, and PT-24).

Biowall process monitoring is being conducted at two locations to determine if, and when, any biowall maintenance activities should be performed. The first location is within Biowalls B1/B2 (MWT-27 and MWT-28) in the segment that runs along the pilot-scale biowalls that were installed in July 2005. The second location is within Biowall C2 (MWT-23), the furthest downgradient biowall. The objectives of biowall process monitoring for operations and maintenance (O&M) activities are as follows:

- Monitor the long-term performance and sustainability of the biowalls;
- Monitor substrate depletion and geochemical conditions under which the effectiveness of the biowalls may decline; and
- Determine if, and when, the biowalls need maintenance (i.e., need to be recharge with additional organic substrate).

To monitor the effectiveness of the biowalls and achieve the objectives above, LTM will continue under this TO on a semi-annual basis. Water level measurements will be collected from all 34 existing monitoring wells at the Ash Landfill; however, as described above, only the plume performance, biowall process, and off-site monitoring well (14 in total) will have groundwater samples analyzed (**Figure 10.5**) (**Worksheet #18**). A low-flow bladder pump will be used to purge the wells, to minimize disturbances and limit turbidity. The wells will be purged and samples will be collected in accordance with SOP ENV-02 (**Worksheet #21**).

The performance of the biowalls will be monitored through the use of lines-of-evidence. COC concentrations and geochemical parameters from wells within the biowalls are closely monitored and compared to EPA benchmarks of parameters which promote natural attenuation of chlorinated solvents (**Table 17.3**) (EPA, 1998). If groundwater COC concentrations and groundwater chemistry within the biowalls diverge from several of these benchmarks then biowalls recharge will be considered. The same benchmark values (EPA, 1998) will be used to determine if the environment is conducive to reductive dechlorination and the effectiveness of MNA.

Table 17.3 – Biowall Performance Benchmark Values

PARAMETER	VALUE	NOTES
Dissolved Oxygen (DO)	• <1 mg/L	
Oxidation Reduction Potential (ORP)	• < 50 mV	
Total Organic Content (TOC)	• > 20 mg/L	
Sulfate	• < 20 mg/L	• Consumed compared to upgradient
Ethane / Ethene	• > 0.1 mg/L	
Methane	• > 0.5 mg/L	
Manganese	• --	• Elevated when compared to upgradient
Ferrous Iron (Fe 2+)	• > 1 mg/L	• Elevated when compared to upgradient
Trichloroethene (TCE)	• Non-Detect (ND)	• ND
DCE	• Low Conc.	• ND or low concentrations (daughter product of TCE breakdown)
VC	• Low Conc.	• ND or low concentrations (daughter product of DCE breakdown)

The analytes and the respective analysis to be used at the Ash Landfill are summarized below:

- VOCs – EPA SW846 Method 8260C
- Sulfate – EPA Method 300.0
- Methane, ethane, ethene (MEE) – RSK 175
- Total organic carbon (TOC) – EPA SW846 Method 9060A

In addition, a Hach® DR/850 colorimeter will be used in the field to measure manganese and ferrous iron at select wells (Worksheet #18). Manganese and ferrous iron will be measured by EPA Method 8034 and 8146, respectively. The same geochemical parameters presented in **Table 17.2** will be recorded throughout the purging process. A comprehensive list showing analyses to be performed at each well is included on **Worksheet #18**.

SEAD 16/17

As described on **Worksheet #10**, LTM at the SEAD-16/17 has been ongoing since December 2007 since the RA was completed to excavate and dispose of contaminated soils with select metals and PAHs. As noted in the ROD for SEAD-16/17, the groundwater is to be assessed until the applicable cleanup standards are achieved before any LUCs are to be lifted to allow unlimited exposure and unrestricted use.

To determine when the applicable cleanup standards are met (**Worksheet #15**), LTM will continue under this TO on an annual basis. Water level measurements will be measured and groundwater samples will be collected from the six

existing monitoring wells at SEAD-16 and the five existing monitoring wells at SEAD-17 (**Figure 10.6** and **10.7**). A low-flow peristaltic pump will be used to purge the wells, and the geochemical parameters presented in **Table 17.2** will be recorded throughout the purging process. The wells will be purged and samples will be collected in accordance with SOP ENV-02 (**Worksheet #21**).

The groundwater samples will be analyzed for total target analyte list (TAL) metals (except mercury) using analytical method EPA SW846 Method 6020A and total mercury using analytical method EPA SW846 7470A. A comprehensive list showing analyses to be performed at each well is included on **Worksheet #18**.

SEAD-122E, SEAD-25, and SEAD-26

No long-term monitoring was conducted at SEAD-122E and PFAS has not been sampled at any of the three sites. At SEAD-122E, a total of 23 temporary groundwater sampling locations are proposed as shown on **Figure 10.9**;

- One location along each side of the four fuel pads (4 locations per fuel pad)
- One location on each side of the runway, and;
- Three locations along the western side of the runway. The additional three wells along the western side of the runway are proposed to capture downgradient groundwater flow from SEAD-122E.

Up to an additional 10 locations may be installed at SEAD-122E based on regulatory input for a total of 37 potential geoprobe locations.

At SEAD-26, one temporary groundwater sampling location will be advanced on each side of the SEAD (north, east, south, and west side) for a total of four locations as shown on **Figure 10.9**.

At SEAD-25, groundwater grab samples will be collected using existing 12 monitoring wells (**Figure 10.4**).

Groundwater concentrations will be reviewed for PFAS COCs (**Worksheet #17**) and compared with EPA temporary provisional health advisory level (**Worksheet #15**). A Site Investigation report will be written and will include evaluation of potential contamination and recommendations for future actions.

The wells will be purged and samples will be collected in accordance with SOP ENV-02 (**Worksheet #21**) and PFAS SOPs (**Appendix D**). The groundwater samples will be analyzed for PFAS using analytical method EPA SW846 Method 537 (**Table 15.9**). A comprehensive list showing analyses to be performed at each well is included on **Worksheet #18**.

17.2.4 LUC INSPECTIONS

OB Grounds

In addition, the quantitative analytical groundwater sampling being conducted, the vegetative compacted soils placed as part of the remedy and Reeder Creek are to be visually inspected as part of the LTM at the OB Grounds. The inspections are to be done on an annual basis, in conjunction with the groundwater sampling activities.

Visual inspections of the vegetative compacted soil cover placed over the contaminated soils at the OB Grounds is required to assure the long-term integrity of the soil cover, including the potential mobilization and migration of lead-contaminated soil buried beneath the cover. Additionally, the soil caps serve to prevent direct contact with, and incidental ingestion of soils containing lead concentrations up to 500 mg/kg by terrestrial wildlife at the site. Each vegetative cap area is to be inspected for soil erosion due to fluvial processes, human-induced erosion (e.g., tire ruts) and animal burrows or trails. Any disturbances are to be documented and addressed, if necessary to prevent potential exposure or migration of contaminated soils. Additionally, any large trees and shrubs will be documented and recommended for removal. Inspection forms are provided in Appendix B.

Additionally, the LTM program at the OB Grounds includes a qualitative assessment (i.e., visual inspection) of Reeder Creek for evidence of migration of material via surface water flow or groundwater transport of contaminants into the remediated section of Reeder Creek adjacent to and down gradient of the OB Grounds. The visual inspection consists of walking the creek bed (or embankment) to look for evidence of soil erosion or sloughing from the Creek embankment adjacent to the OB Grounds and/or the accumulation of sediment along the stream bed. Presently, quantitative

monitoring of sediment quality (i.e., submitting samples for copper and lead analysis as identified in the approved remedy for the Site in the ROD) is not included as part of the LTM activities; the U.S. Army Corps of Engineers (Army), the U.S. Environmental Protection Agency (EPA), and the New York State Department of Environmental Conservation (NYSDEC) agreed that until data indicated that either groundwater transport of contaminants or soil transport from the OB Grounds was occurring, sampling and analysis of Creek sediments would not be required.

SEAD-25

The remedy for SEAD-25 (**Worksheet #10**) required the implementation and maintenance of LUCs. The LUC requirements are detailed in the Final Record of Decision for SEAD-25 and SEAD-26 (Parsons 2004), Addendum 1 in the *Land Use Control Remedial Design for SEAD 27, 66, 64A, Final* (2006) and are additionally covered under the area-wide LUCs Planned Industrial/Office or Warehousing Area ("PID Area") (Parsons, 2004; 2006). The selected LUCs for SEAD-25 are as follows:

- Prevent residential housing, elementary and secondary schools, childcare facilities and playground activities; and
- Prevent access to and use of groundwater at SEAD-25, for purposes other than required monitoring, until NYS Class GA Groundwater Standards are met.

As part of the LTM program, SEAD-25 will continue to be inspected to determine if the LUCs are being maintained (**Appendix B**).

Ash Landfill

As part of the remedy for the Ash Landfill (**Worksheet #10**), a 12-inch vegetative cover over the Ash Landfill and the Non-Combustible Fill Landfill (NCFL) was constructed to prevent ecological receptors from coming into direct contact with the underlying soils that are contaminated with metals and PAHs. As part of the on-going LTM program, in conjunction with the semi-annual monitoring, the vegetative covers will be inspected to monitor the integrity of the covers and ensure that they have not been disturbed to expose the underlying soil. Each vegetative cap area is to be inspected for soil erosion due to fluvial processes, human-induced erosion (e.g., tire ruts) and animal burrows or trails. Any disturbances are to be documented and addressed, if necessary to prevent potential exposure or migration of contaminated soils. Additionally, any large trees and shrubs will be documented and recommended for removal. Inspection forms will be used to record this information for each vegetative cap area. Field forms are provided in **Appendix B**.

SEAD-16/17

The ROD for SEAD-16/17 also required the implementation, maintenance, inspection, and periodic reporting of LUCs prohibiting the use of the land at the AOCs for residential purposes. The LUCs for SEAD-16/17 are as follows:

- Prevent access to or use of groundwater until NYS Class GA groundwater standards are achieved; and
- To prohibit residential housing, elementary and secondary schools, child care facilities, and playground activities at the sites.

As part of the LTM program, SEAD-16/17 will continue to be inspected to determine if the LUCs are being maintained (**Appendix B**).

SEAD-122E and SEAD-26

No additional LUC inspections will be performed at these sites.

17.2.5 DEMOBILIZATION

Upon completion of the field activities, all equipment and materials will be packaged and removed from the site. The samples will be packaged in coolers and shipped to the analytical laboratory as described in **Worksheets #26** and **#27**. All field documentation will be electronically scanned and the rental sampling equipment will be returned to the vendor. The field office shall be cleaned and organized to facilitate efficient sampling preparation during the next field event.

Worksheet #18: Sampling Locations and Methods

(EPA UFP-QAPP Guidance Manual, Section 3.1.1 and 3.1.2, EPA Guidance 2106-G-05 Section 2.3.1 and 2.3.2)

The sample rationale for the LTM at the OB Grounds, SEAD-25, Ash Landfill, and SEADs 16/17 is summarized in the **Table 18.1**. Monitoring well locations for groundwater sample collections are shown in **Figures 10.2 through 10.7**. Sample ID nomenclature is explained in **Worksheet #26**.

Table 18.1 – Sampling Locations and Methods for the OB Grounds

LOCATION ID	SAMPLE ID(1)	MATRIX	SCREEN DEPTH (FT BGS)	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
MW23-1	OBLM0071	GW	7-12	Sample	Total Cu and Total Pb / Metals	ENV-02	
MW23-1	OBLM0071MS	GW	7-12	Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Total Cu and Total Pb / Metals	ENV-02	See note #2
MW23-1	OBLM0071MSD	GW	7-12	MS/MSD	Total Cu and Total Pb / Metals	ENV-02	See note #2
MW23-1	OBLM0072	GW	7-12	Field Duplicate	Total Cu and Total Pb / Metals	ENV-02	See note #2
MW23-2	OBLM0073	GW	7-12	Sample	Total Cu and Total Pb / Metals	ENV-02	
MW23-3	OBLM0074	GW	7-12	Sample	Total Cu and Total Pb / Metals	ENV-02	
MW23-4	OBLM0075	GW	9.5-14.5	Sample	Total Cu and Total Pb / Metals	ENV-02	
MW23-5	OBLM0076	GW	9.5-14.5	Sample	Total Cu and Total Pb / Metals	ENV-02	
MW23-6	OBLM0077	GW	9.5-14.5	Sample	Total Cu and Total Pb / Metals	ENV-02	
N/A	OBLM00106	Aqueous	--	Rinse Blank	Total Cu and Total Pb / Metals		If disposable equipment is not used

Key: GW = groundwater; Cu = Copper; Pb = Lead

(1) For database consistency, Round 11 sample ID sequence will begin with OBLM0071 which is one increment higher than the last sample in the previous LTM event (Round 10).

(2) MS/MSD and Field Duplicate are moved sequentially for each round. In the LTM round subsequent to the example shown, the MS/MSD and Field Duplicate will be collected at location MW23-2, as groundwater conditions allow.

Table 18.2 – Sampling Locations and Methods for SEAD-25

LOCATION ID	SAMPLE ID(1)	MATRIX	SCREEN DEPTH (FT BGS)	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
MW25-2	25LM20124	GW	3.4-7.4	Sample	VOCs Nitrate/Nitrite Sulfate/Chloride Methane/Ethene/Ethane (MEE) Sodium/Iron Sulfide[3]	ENV-02	
MW25-3	25LM20125	GW	4-6	Sample		ENV-02	
MW25-3	25LM20125MS	GW	4-6	MS/MSD		ENV-02	See note #2
MW25-3	25LM20125MSD	GW	4-6	MS/MSD		ENV-02	See note #2
MW25-3	25LM20126	GW	4-6	Field Duplicate		ENV-02	See note #2
MW25-9	25LM20127	GW	3.2-4.0	Sample		ENV-02	
MW25-10	25LM20128	GW	3.2-5.2	Sample		ENV-02	
MW25-17	25LM20129	GW	4.6-9.1	Sample		ENV-02	
N/A	25LM00114	Aqueous	--	Rinse Blank			If disposable equipment is not used
N/A	25LM00030	Aqueous	--	Trip Blank	VOCs		One trip blank per cooler shipment

Key: GW = groundwater

(1) For database consistency, Year 10 sample ID sequence will begin with 25LM20124 which is one increment higher than the last sample in the previous LTM event (Year 9).

(2) MS/MSD and Field Duplicate are moved sequentially for each round. In the LTM round subsequent to the example shown, the MS/MSD and Field Duplicate will be collected at location MW23-2, as groundwater conditions allow.

(3) Field test performed for sulfide at each well using Hach colorimeter.

Table 18.3 – Sampling Locations and Methods for the Ash Landfill

LOCATION ID	SAMPLE ID(1)	MATRIX	SCREEN DEPTH (FT BGS)	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
PT-17	ALBW20375	GW		Sample	VOC, Sulfate, TOC, MEE, Fe, Mn	ENV-02	Fe, Mn are field tests
PT-18A	ALBW20376	GW	4.8-9.8	Sample	VOC	ENV-02	
PT-22	ALBW20377	GW		Sample	VOC	ENV-02	
PT-24	ALBW20378	GW		Sample	VOC	ENV-02	
MWT-7	ALBW20379	GW		Sample	VOC, Sulfate, TOC, MEE, Fe, Mn	ENV-02	Fe, Mn are field tests
MWT-22	ALBW20380	GW	7.5-12.5	Sample	VOC	ENV-02	
MWT-24	ALBW20381	GW		Sample	VOC	ENV-02	
MWT-25	ALBW20382	GW		Sample	VOC	ENV-02	
MW-56	ALBW20383	GW		Sample	VOC	ENV-02	
MWT-23	ALBW20384	GW		Sample	VOC, Sulfate, TOC, MEE, Fe, Mn	ENV-02	Fe, Mn are field tests
MWT-26	ALBW20385	GW		Sample	VOC, Sulfate, TOC, MEE, Fe, Mn	ENV-02	Fe, Mn are field tests
MWT-27	ALBW20386	GW		Sample	VOC, Sulfate, TOC, MEE, Fe, Mn	ENV-02	Fe, Mn are field tests
MWT-28	ALBW20387	GW		Sample	VOC, Sulfate, TOC, MEE, Fe, Mn	ENV-02	Fe, Mn are field tests
MWT-28	ALBW20387MS	GW		MS/MSD	VOC, Sulfate, TOC, MEE, Fe, Mn	ENV-02	See note #2. Fe, Mn are field tests
MWT-28	ALBW20387MSD	GW		MS/MSD	VOC, Sulfate, TOC, MEE, Fe, Mn	ENV-02	See note #2. Fe, Mn are field tests
MWT-28	ALBW20388	GW		Duplicate	VOC, Sulfate, TOC, MEE, Fe, Mn	ENV-02	See note #2. Fe, Mn are field tests
MWT-29	ALBW20389	GW		Sample	VOC, Sulfate, TOC, MEE, Fe, Mn	ENV-02	Fe, Mn are field tests
MW-40	ALBW20390	GW	7.5-14.5	Sample	Fe, Mn	ENV-02	Field analysis, not submitted to lab
N/A	ALBW00126	Aqueous	--	Rinse Blank	VOC, Sulfate, TOC, MEE		
N/A	ALBW00049	Aqueous	--	Trip Blank	VOC		
N/A	ALBW00050	Aqueous	--	Trip Blank	VOC		

Key: GW = groundwater; Fe = Iron; Mn = Manganese

(1) For database consistency, Round 22 sample ID sequence will begin with ALBW20375 which is one increment higher than the last sample in the previous LTM event (Round 21).

(2) MS/MSD and Field Duplicate are moved sequentially for each round between biowall locations MWT-23, MWT-27, and MWT-28. In the LTM round subsequent to the example shown, the MS/MSD and Field Duplicate will be collected at location MWT-23, as groundwater conditions allow.

(3) MW-40 is sampled in the field for Mn and Fe and used as a background comparison of Mn and Fe for the biowall and plume performance wells.

Table 18.4 – Sampling Locations and Methods for SEAD-16

LOCATION ID	SAMPLE ID(1)	MATRIX	SCREEN DEPTH (FT BGS)	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
MW16-1	16LM20056	GW	3.3-5.3	Sample	TAL Metals	ENV-02	
MW16-1	16LM20056MS	GW	3.3-5.3	MS/MSD		ENV-02	See note #2
MW16-1	16LM20056MSD	GW	3.3-5.3	MS/MSD		ENV-02	See note #2
MW16-1	16LM20057	GW	3.3-5.3	Field Duplicate		ENV-02	See note #2
MW16-2	16LM20058	GW	1.4-3.4	Sample		ENV-02	
MW16-4	16LM20059	GW	2.3-4.3	Sample		ENV-02	
MW16-5	16LM20060	GW	2.5-4.5	Sample		ENV-02	
MW16-6	16LM20061	GW	1.3-3.3	Sample		ENV-02	
MW16-7	16LM20062	GW	2.6-4.6	Sample		ENV-02	
N/A	16LM00104	Aqueous	--	Rinse Blank			If disposable equipment is not used

Key: GW = groundwater

(1) For database consistency, Round 9 sample ID sequence will begin with 16LM20056 which is one increment higher than the last sample in the previous LTM event (Round 8).

(2) MS/MSD and Field Duplicate are moved sequentially for each round. In the LTM round subsequent to the example shown, the MS/MSD and Field Duplicate will be collected at location MW16-2, as groundwater conditions allow.

(3) If SEAD 16 and SEAD 17 are sampled concurrently, an additional MS/MSD, duplicate, and rinse blank (if applicable) are not needed at SEAD-17.

Table 18.5 – Sampling Locations and Methods for SEAD-17

LOCATION ID	SAMPLE ID(1)	MATRIX	SCREEN DEPTH (FT BGS)	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
MW17-1	17LM20040	GW	3.4-7.4	Sample	TAL Metals	ENV-02	
MW17-2	17LM20041	GW	3.3-5.3	Sample		ENV-02	
MW17-3	17LM20042	GW	3.1-5.1	Sample		ENV-02	
MW17-4	17LM20043	GW	3.1-5.1	Sample		ENV-02	
MW17-5	17LM20044	GW	3.4-7.9	Sample		ENV-02	

Key: GW = groundwater

(1) For database consistency, Round 9 sample ID sequence will begin with 17LM20040 which is one increment higher than the last sample in the previous LTM event (Round 8).

(2) If SEAD 16 and SEAD 17 are sampled concurrently, an additional MS/MSD and duplicate are not needed at SEAD-17.

Table 18.6 – Sampling Locations and Methods for PFAS at SEAD-122E

LOCATION ID	SAMPLE ID(1)	MATRIX	SCREEN DEPTH (FT BGS)5	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
TMW-122E-1	122SI20001	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-2	122SI20002	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-3	122SI20003	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-4	122SI20004	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-5	122SI20005	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-6	122SI20006	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-7	122SI20007	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-8	122SI20008	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-9	122SI20009	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-10	122SI20010	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-11	122SI20011	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-12	122SI20012	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-13	122SI20013	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-14	122SI20014	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-15	122SI20015	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-16	122SI20016	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-17	122SI20017	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-18	122SI20018	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-19	122SI20019	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-20	122SI20020	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-21	122SI20021	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-22	122SI20022	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
TMW-122E-23	122SI20023	GW	N/A	Sample	PFAS	ENV-02, Appendix D	
N/A	122SI00001	Aqueous	N/A	Equipment Blank	PFAS	ENV-02, Appendix D	One equipment blank per day. 2
N/A	122SI01000	Aqueous	N/A	Field Blank	PFAS	ENV-02, Appendix D	One field blank per day. 3
N/A	122SI00100	Aqueous	N/A	Trip Blank	PFAS	ENV-02, Appendix D	One trip blank per cooler shipment4

Key: GW = groundwater

(1) Two MS/MSD and three field duplicates will be collected. The locations of the MS/MSD and field duplicate will be determined in the field based on site conditions. One of the existing sample IDs in the table above will be appended with MS and MSD (e.g., 122SI20001MS and 122SI20001MSD). Each set of MS/MSD samples will have a low and moderate spike. The field duplicate will be collected at the same locations as the MS/MSD, with the exception of one. Field duplicate sample ID will be one larger than the last ID shown in the table (e.g., 122SI200024).

(2) One equipment (rinse) blank will be collected per day of sampling. Each day the equipment (rinse) blank ID will increase by one.
(3) One field blank will be collected per day of sampling. Each day the field blank ID will increase by one.
(4) One trip blank will be included per cooler of samples shipped. Each trip blank will increase by one.
(5) New locations will be drilled so no screen depth is known at this time.

Table 18.7 – Sampling Locations and Methods for PFAS at SEAD-25

LOCATION ID	SAMPLE ID(1)	MATRIX	SCREEN DEPTH (FT BGS)	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
MW25-1	25SI20001	GW	4-6	Sample	PFAS	ENV-02, Appendix D	
MW25-2	25SI20002	GW	3.4-7.4	Sample	PFAS	ENV-02, Appendix D	
MW25-3	25SI20003	GW	4-6	Sample	PFAS	ENV-02, Appendix D	
MW25-6	25SI20004	GW	3.2-5.2	Sample	PFAS	ENV-02, Appendix D	
MW25-8	25SI20005	GW	3.2-4.0	Sample	PFAS	ENV-02, Appendix D	
MW25-9	25SI20006	GW	3.2-4.0	Sample	PFAS	ENV-02, Appendix D	
MW25-10	25SI20007	GW	3.2-5.2	Sample	PFAS	ENV-02, Appendix D	
MW25-13	25SI20008	GW	2.7-3.5	Sample	PFAS	ENV-02, Appendix D	
MW25-15	25SI20009	GW	3.9-5.4	Sample	PFAS	ENV-02, Appendix D	
MW25-17	25SI20010	GW	4.6-9.1	Sample	PFAS	ENV-02, Appendix D	
MW25-18	25SI20011	GW	4.4-8.9	Sample	PFAS	ENV-02, Appendix D	
MW25-19	25SI20012	GW	5.3-9.8	Sample	PFAS	ENV-02, Appendix D	
N/A[2]	25SI00001	Aqueous	--	Equipment Blank	PFAS	ENV-02, Appendix D	One equipment blank per day.[2]
N/A[3]	25SI01000	Aqueous	--	Field Blank	PFAS	ENV-02, Appendix D	One field blank per day.[3]
N/A[4]	25SI00100	Aqueous		Trip Blank	PFAS	ENV-02, Appendix D	One trip blank per cooler shipment[4]

Key: GW = groundwater

(1) One MS/MSD and two field duplicates will be collected. The locations of the MS/MSD and field duplicate will be determined in the field based on site conditions. One of the existing sample IDs in the table above will be appended with MS and MSD (e.g., 25SI20001MS and 25SI200001MSD). The field duplicates will be collected at the same location as the MS/MSD and the sample ID will be one larger than the last ID shown in the table (e.g., 25SI20013).

(2) One equipment (rinse) blank will be collected per day of sampling. Each day the equipment (rinse) blank ID will increase by one.

(3) One field blank will be collected per day of sampling. Each day the field blank ID will increase by one.

(4) One trip blank will be included per cooler of samples shipped. Each trip blank will increase by one.

Table 18.8 – Sampling Locations and Methods for PFAS at SEAD-26

LOCATION ID	SAMPLE ID(1)	MATRIX	SCREEN DEPTH (FT BGS)	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
TMW-26-1	26SI20001	GW		Sample	PFAS	ENV-02, Appendix D	
TMW-26-2	26SI20002	GW		Sample	PFAS	ENV-02, Appendix D	
TMW-26-3	26SI20003	GW		Sample	PFAS	ENV-02, Appendix D	
TMW-26-4	26SI20004	GW		Sample	PFAS	ENV-02, Appendix D	
N/A2	26SI00001	Aqueous	--	Equipment Blank	PFAS	ENV-02, Appendix D	One equipment blank per day. 2
N/A3	26SI01000	Aqueous	--	Field Blank	PFAS	ENV-02, Appendix D	One field blank per day. 3
N/A4	26SI00100	Aqueous		Trip Blank	PFAS	ENV-02, Appendix D	One trip blank per cooler shipment4

Key: GW = groundwater

(1) One MS/MSD and one field duplicate will be collected. The locations of the MS/MSD and field duplicate will be determined in the field based on site conditions. One of the existing sample IDs in the table above will be appended with MS and MSD (e.g., 26SI20001MS and 26SI200001MSD). The field duplicates will be collected at the same location as the MS/MSD and the sample ID will be one larger than the last ID shown in the table (e.g., 26SI20005).

(2) One equipment (rinse) blank will be collected per day of sampling. Each day the equipment (rinse) blank ID will increase by one.

(3) One field blank will be collected per day of sampling. Each day the field blank ID will increase by one.

(4) One trip blank will be included per cooler of samples shipped. Each trip blank will increase by one.

Worksheets #19 & 30: Sample Containers, Preservation, and Hold Times

(EPA UFP-QAPP Guidance Manual, Section 3.1.1)

This worksheet summarizes the analytical methods for each sampling matrix, including the required sample volume, containers, preservation, and holding time requirements. Details concerning sampling handling are included on **Worksheets #26 & 27**. All samples will be delivered to Katahdin Analytical Services, located in Scarborough, ME with ice via UPS or FedEx next day delivery.

ANALYTE/ ANALYTICAL GROUP	MATRIX	METHOD/SOP REFERENCE (1)	ACCREDITATION EXPIRATION DATE	CONTAINERS (NUMBER, SIZE, AND TYPE) (2)	PRESERVATION REQUIREMENTS	PREPARATION HOLDING TIME	ANALYTICAL HOLDING TIME	DATA PACKAGE TURNAROUND
VOCs	GW	5030, 8260C / CA-202	01 Feb 2019	3, 40-ml VOA vials w/ PTFE-faced silicone septum	4 ± 2°C, HCl, pH <2	14 days	14 days	21 days
MEE	GW	RSK-175 /CA-336	01 Feb 2019	3, 40-ml VOA vials w/ PTFE-faced silicone septum	4 ± 2°C, HCl, pH <2	14 days	14 days	21 days
Nitrate and Nitrite	GW	353.2 / CA-728	01 Feb 2019	1, 125-ml polyethylene bottle	4 ± 2°C , H2SO4, pH <2	~~28 days~~ 48 hours	28 days	21 days
Chloride and Sulfate	GW	300.0 / CA-742	01 Feb 2019	1, 125-ml polyethylene bottle	4 ± 2°C	28 days	28 days	21 days
Iron, Sodium, Copper, and Lead	GW	6010C / CA-608	01 Feb 2019	1, 250-ml polyethylene bottle	4 ± 2°C, HNO3, pH <2	6 months	6 months	21 days
TAL Metals, excluding Hg	GW	6020A / CA-627	01 Feb 2019	1, 250-ml polyethylene bottle	4 ± 2°C, HNO3, pH <2	6 months	6 months	21 days
Mercury (3)	GW	7470A /CA-615	01 Feb 2019	1, 250-ml polyethylene bottle	4 ± 2°C, HNO3, pH <2	28 days	28 days	21 days
TOC	GW	9060A / CA-763	01 Feb 2019	3, 40-ml VOA vials w/ PTFE-faced silicone septum	4 ± 2°C, H2SO4, pH <2	28 days	28 days	21 days
PFAS	GW	537_Modified / WS-LC-0025	31 Jan 2017	2, 250 ml HDPE bottles	4 ± 2°C;	7 days	40 days	21 days

(1) Laboratory SOPs (Appendix C) are subject to revision and updates during duration of the project, lab will use the most current revision of the SOP at the time of analysis.
(2) Sample size is a minimum, the containers listed will be filled to compensate for any required re-analysis or re-extractions. For samples requiring Matrix Spike/Matrix Spike Duplicate (MS/MSD) containers listed should be tripled.
(3) Mercury samples are to be extracted from TAL metals sample container.

Worksheet #20: Field Quality Control

(EPA UFP-QAPP Guidance Manual, Section 3.1.1)

This worksheet summarizes the QC samples to be collected and analyzed for the project. It shows the relationship between the number of field samples and associated QC samples for each combination of analyte/analytical group and matrix. Note if samples are collected over the estimated number shown, additional QC samples will be collected at the rate shown.

Table 20.1 – LTM Field and Quality Control Samples

SITE	MATRIX	ANALYTICAL GROUP	ESTIMATED NO. OF FIELD SAMPLES	TRIP BLANK (FOR VOC ONLY)	EQUIPMENT BLANK	FIELD DUPLICATES	MATRIX SPIKE / MATRIX SPIKE DUPLICATES	ESTIMATED NUMBER OF TOTAL ANALYSES
OB Grounds	Groundwater	All parameters	6	N/A	N/A[1]	10%	5%	8
SEAD 25			5	1 per cooler	1 per week			9
Ash Landfill			14	1 per cooler	1 per week			19
SEAD 16/17			11	N/A	N/A[1]			13

(1) Dedicated tubing or disposable equipment is used at OB Grounds and SEAD-16/17 therefore equipment blanks are not applicable.

Table 20.2 – PFAS Field and Quality Control Samples

SITE	MATRIX	ANALYTICAL GROUP	ESTIMATED NO. OF FIELD SAMPLES	TRIP BLANK	FIELD BLANK	EQUIPMENT BLANK	FIELD DUPLICATES	MATRIX SPIKE / MATRIX SPIKE DUPLICATES	ESTIMATED NUMBER OF TOTAL ANALYSES
SEAD-122E	Groundwater	PFAS	23	1 per cooler	1 per day	1 per day	10%	5%	31
SEAD 25			12	1 per cooler	1 per day	1 per day			18
SEAD-26			4	1 per cooler	1 per day	1 per day			9

(1) If samples are collected from more than one site per day, the field and equipment blank may be shared between sites.

Worksheet #21: Field Standard Operating Procedures

(EPA UFP-QAPP Guidance Manual, Section 3.1.2)

The field SOPs to be used during the investigation are listed in the below table. Copies of these field SOPs are provided in **Appendix A**.

REFERENCE NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	SOP ORIGINATING ORGANIZATION	RELATED EQUIPMENT TYPES	MODIFIED FOR PROJECT? (Y/N)	COMMENTS
SOP CHEM-01	Chemistry Data Management May 20, 2015 Revision #00	Parsons	None	N	See Appendix A
SOP ENV-02	Groundwater Sampling February 18, 2015 Revision #00	Parsons	Sampling tool(s), sample containers	N	See Appendix A
Appendix D	PFAS Groundwater Sampling November 29, 2016 Revision #00	Parsons	Sampling tool(s), sample containers	Y	See Appendix D

Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection

(EPA UFP-QAPP Guidance Manual, Section 3.1.2.4)

This worksheet describes the field equipment needed for the project and the associated calibration, maintenance, testing, and inspection procedures for that field equipment. This worksheet also documents the field equipment's frequency of activity, acceptance criteria, and corrective action requirements.

FIELD EQUIPMENT	CALIBRATION, VERIFICATION, TESTING, OR MAINTENANCE ACTIVITY	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	RESPONSIBLE PERSON	SOP REFERENCE (1)
Horiba U-52	Calibration and Visual inspection	Daily, prior to sampling	Standard calibration solutions for pH, ORP, and Spec. Cond.	Recalibrate or use alternative equipment	Sample Team Lead	Operator Manual (Appendix F)
YSI DO Meter	Calibration and Visual inspection	Daily, prior to sampling	Standardize DO to atmospheric pressure	Recalibrate or use alternative equipment	Sample Team Lead	Operator Manual (Appendix F)
Turbidity Meter	Calibration and Visual inspection	Daily, prior to sampling	Standard calibration solutions for NTU	Recalibrate or use alternative equipment	Sample Team Lead	Operator Manual (Appendix F)
Bladder Pump	Visual inspection, Maintenance, and Decontamination	Between sampling locations	Equipment is clean	Decontaminate bladder pump, replace bladder, seals, and tubing prior to sampling	Sample Team Lead	ENV-02
Peristaltic Pump	Visual inspection, Maintenance, and Decontamination	Between sampling locations	Equipment is clean	Replace tubing prior to sampling	Sample Team Lead	ENV-02, ENV-03
Hach colorimeter	Visual inspection	Daily, prior to sampling	Equipment is clean, reagents powder pillows are dry and pillows are not damaged	Decontaminate sample containers, use a new reagent pillow or order new reagent	Sample Team Lead	Operator Manual (Appendix F)

(1) See Project SOP Reference Table (**Worksheet #21**) for SOP titles.

Worksheet #23: Analytical Standard Operating Procedures

(EPA UFP-QAPP Guidance Manual, Section 3.2.1)

The applicable SOPs to be used for analysis of samples collected during the investigation are listed in the below tables. The laboratory SOP references were provided by Katahdin and are presented in **Appendix C**.

SOP #	TITLE, DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX/ ANALYTICAL GROUP	SOP OPTION OR EQUIPMENT TYPE	MODIFIED FOR PROJECT?
CA-615	Digestion and Analysis of Aqueous Samples for Mercury by USEPA Method 7470A, 06/14, Revision 8.	Definitive	Groundwater/Metals	Mercury Analyzer	N
CA-604	Acid Digestion of Aqueous Samples by EPA Method 3010A for ICP and ICP-MS analysis of Total or Dissolved Metals, 06/16, Revision 7.	Definitive	Groundwater/Metals	NA	N
CA-627	Trace Metals Analysis By ICP-MS Using USEPA Method 6020A, 06/16, Revision 11.	Definitive	Groundwater/Metals	ICPMS	N
CA-728	Total Nitrate/Nitrite, Nitrite & Nitrate With Cadmium Reduction By Automated Colorimetry, 05/12, Revision 8.	Definitive	Groundwater/Nitrate, Nitrite	LACHAT 10-107-4-1C	N
CA-742	Anions by Ion Chromatography (IC) – Method 300.0, 08/15, Revision 10.	Definitive	Groundwater/Chloride, Sulfate	IC	N
CA-763	Analysis of TOC, DOC, and TIC in Aqueous Samples using the Shimadzu Carbon Analyzer: EPA Method 415.2, SW846 9060A and SM5310B, 07/14, Revision 8.	Definitive	Groundwater/TOC	TOC Analyzer	N
CA-202	Analysis of VOAs by Purge and Trap GC/MS: SW-846 Method 8260, 07/16, Revision 16	Definitive	Groundwater/VOCs	GC/MS	N
CA-336	Dissolved Gas Analysis in Water Samples Using GC Headspace Equilibration Technique EPA SOP RSK-175, 05/13, Revision 7	Definitive	Groundwater/Dissolved Gases	GC	N
CA-608	Trace Metals Analysis by ICP-AES Using USEPA Method 6010, 07/16, Revision 17	Definitive	Groundwater/Metals	ICP-AES	N
WS-LC-0025	Perfluroalkyl Substances (PFAS) in Water, Soils, Sediments and Tissue by LC/MS/MS, 12/09/16, Revision 2.1	Definitive	Groundwater/PFAS	LC/MS/MS	N

Worksheet #24: Analytical Instrument Calibration

(EPA UFP-QAPP Guidance Manual, Section 3.2.2)

The Analytical Instrument Calibration Table and the specific analytical method SOP references are provided in **Appendix C**.

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
GC/MS	Instrument tune check	NA	Prior to ICAL and prior to each 12-hour period of sample analysis	Specific ion abundance criteria of BFB from method.	Retune instrument and verify	Analyst, Department Manager	CA-202
GC/MS	Initial Calibration (ICAL) - Five-point initial calibration is required for all VOCs.	1.0 – 200 ug/ml	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: • Option 1: RSD for each analyte = 15%; • Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; • Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Analyst, Department Manager	CA-202
GC/MS	Second Source Calibration Verification (ICV)	NA	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	CA-202
GC/MS	Continuing Calibration (CCV)	NA	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; t	Analyst, Department Manager	CA-202
GC/FID	ICAL - Five-point initial calibration is required for all VOCs.	Methane: 0.005 – 1.197 ug/ml	Instrument receipt, major instrument change, when	Average %RSD must be ≤20	Recalibrate and/or perform necessary equipment maintenance. Check calibration	Analyst, Department Manager	CA-336

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
		Ethane: 0.009-2.245 ug/ml Ethane: 0.009-20.95	CCV does not meet criteria		standards. Reanalyze affected data.		
GC/FID	ICV	NA	Immediately following ICAL, analysis of a second source standard prior to sample analysis.	The %D of the expected value must be ≤20% for all target analytes. All target analytes within established RT windows.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	CA-336
GC/FID	CCV	NA	If initial calibration analyzed, daily and after 10 field samples, and at end of sequence.	%D for all target analytes within 20% All target analytes within established RT windows.	Evaluate the samples: If the %RPD >20% and sample results are < PQL, narrate. If %RPD >20% and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples after last acceptable CV.	Analyst, Department Manager	CA-336
Lachat	ICAL – Minimum of a 6-point calibration curve plus a blank is prepared.	0.05 - 2 mg/L	Prior to sample analysis	Linear Regression Correlation Coefficient ≥0.995	• Investigate source of problem • Recalibrate	Analyst, Department Manager	CA-728
Lachat	ICV	NA	Once after each ICAL, prior to beginning a sample run.	%R must within 90%-110% for all	• If the ICV fails high, report samples that are <PQL. • Redigest, recalibrate and/or reanalyze other samples.	Analyst, Department Manager	CA-728
Lachat	CCV	NA	One after every 10 samples	%R must within 90%-110%	• If the CCV fails high, report samples that are <PQL. • Redigest, recalibrate and/or reanalyze other samples back to last acceptable CCV recovery	Analyst, Department Manager	CA-728
IC	ICAL – A minimum of a 5-	Chloride – 01 – 10 mg/L	Prior to sample analysis.	R^2 must be ≥0.995.	Correct problem and rerun calibration.	Analyst, Department Manager	CA-742

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
	point calibration is prepared.	Sulfate – 0.2 – 20 mg/L					
IC	ICV	NA	Once after each ICAL prior to sample analysis.	The %R must be within 90-110% of true value and retention times (RTs) must be within appropriate windows.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	CA-742
IC	CCV	NA	After every 10 samples and at the end of the sequence.	The %R must be within 90-110% of true value and all project analytes must be within established RT windows.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	CA-742
ICP/AES	ICAL - 1 point calibration plus blank	NA	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Analyst, Department Manager	CA-608
ICP/AES	ICV	NA	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	CA-608
ICP/AES	CCV	NA	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst, Department Manager	CA-608
ICP/MS	ICAL - 1 point calibration plus blank	NA	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Analyst, Department Manager	CA-627

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
ICP/MS	ICV	NA	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes, within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	CA-627
ICP/MS	CCV	NA	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst, Department Manager	CA-627
Cold Vapor AA	ICAL - 5 points plus a calibration blank	NA	Daily ICAL prior to sample analysis.	r2 ≥0.99	Correct problem, then repeat ICAL.	Analyst, Department Manager	CA-615
Cold Vapor AA	ICV	NA	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of the true value.	Correct problem. Rerun ICV. If that fails, Rerun ICAL.	Analyst, Department Manager	CA-615
Cold Vapor AA	CCV	NA	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst, Department Manager	CA-615

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
TOC Analyzer	ICAL – Minimum of a 5-point calibration curve plus a blank is prepared.	0.2- 20 mg/L	Initially, when the daily CCV does not pass, but, no longer than every 3 months.	Correlation coefficient, r ≥ 0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Department Manager	CA-763
TOC Analyzer	ICV	NA	Once after each ICAL, prior to beginning a sample run.	90-110%R	(1) If the ICV fails high, report samples that are <PQL. (2) Redigest, recalibrate and/or reanalyze other samples.	Analyst, Department Manager	CA-763
TOC Analyzer	CCV	NA	Every 10 samples and at the end of the run	90-110%R	If the CCV fails high, report samples that are <PQL. Recalibrate and/or reanalyze samples back to last acceptable CCV recovery.	Analyst, Department Manager	CA-763
LC/MS/MS	Tune Check	NA	Prior to ICAL and after any mass calibration or maintenance is performed.	Tuning standard must contain analytes of interest or appropriate substitute. Mass assignments of tuning standard within 0.5 amu of true value.	Retune instrument. If the tuning will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Analyst, Department Manager	WS-LC-0025
LC/MS/MS	ICAL- Minimum 5-point initial calibration for target analytes, lowest concentration standard at or below the reporting limit	0.5- 400 ng/mL	Initial calibration prior to sample analysis	Each calibration point for each analyte must calculate to be within 75-125%, except the lowest cal point which must calculate to within 70-130%.	Evaluate standards, chromatography, and mass spectrometer response. If problem found with above, correct as appropriate, then repeat initial calibration.	Analyst, Department Manager	WS-LC-0025

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
LC/MS/MS	ICV or Second Source Verification (SSV)	NA	Once per initial calibration, following initial calibration.	All reported analytes and labeled compounds within ±25% of true value	Evaluate data. If problem (e.g., concentrated standard, plugged transfer line) found, correct, then repeat SSV. If it still fails, then repeat initial calibration.	Analyst, Department Manager	WS-LC-0025
LC/MS/MS	CCV	NA	Before sample analysis, after every 10 samples, and at the end of the sequence any mass calibration or maintenance is performed.	All reported analytes and labeled compounds within ±25% of true value	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment. For closing CCVs, if compounds are not identified as critical compounds of concern report results with qualifiers. For closing CCVs, if the compound is identified as a critical compound of concern, then recalibrate, and reanalyze all affected samples since the last acceptable CCV; or immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst, Department Manager	WS-LC-0025

Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection

(EPA UFP-QAPP Guidance Manual, Section 3.2.3)

This worksheet provides information on analytical instruments and equipment, maintenance, testing, and inspection. To ensure that the analytical instruments and equipment are available and in working order when needed, all laboratory analytical equipment will undergo maintenance and testing procedure in accordance with the laboratory SOPs (provided in **Appendix C)**.

INSTRUMENT/ EQUIPMENT	MAINTENANCE ACTIVITY	TESTING ACTIVITY	INSPECTION ACTIVITY	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	RESPONSIBLE PERSON	SOP REFERENCE (1)
GC/MS VOCs	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in lab Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-202
GC/FID	Check pressure and gas supply daily. Change septa and/or GC injector glass liner as needed. Replace or cut GC column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	MEE	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-336
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Metals	Torch, nebulizer chamber, pump, pump tubing.	Prior to ICAL and as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-608
ICP-MS	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	TAL Metals	Torch, nebulizer, spray chamber, pump tubing.	Prior to ICAL and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-627

INSTRUMENT/ EQUIPMENT	MAINTENANCE ACTIVITY	TESTING ACTIVITY	INSPECTION ACTIVITY	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	RESPONSIBLE PERSON	SOP REFERENCE (1)
Cold Vapor AA	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Mercury	Tubing, sample probe, optical cell.	Prior to ICAL and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-615
Lachat	Change the pump tubing monthly, replace capillary tubing, clean valves and flow cells.	Nitrate + Nitrite	Pump tubing, capillary tubing, reagent bottles, manifolds	Daily	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-728
TOC Combustion Analyzer	Check level of dilution water, drain vessel water, humidifier water, auto sampler rinse water and phosphoric acid vessel and fill as needed. Replace oxygen cylinder.	Total Organic Carbon	Tubing, sample boat, syringe, humidifier, rinse reservoir, phosphoric acid vessel, oxygen pressure	Prior to initial calibration and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-763
Ion Chromatograph	Check regenerate pump tubing and replace as needed. Clean or regenerate column as needed. Replace analytical column or guard column as needed. Change suppressor as needed.	Chloride, Sulfate	Tubing, column, suppressor.	Prior to initial calibration and/or as necessary.	Passing ICAL or CCV.	Correct problem and repeat calibration or CCV.	Analyst, Department Manager	CA-742
LC/MS/MS	Replace columns as needed, check eluent reservoirs	PFAS	Instrument performance and sensitivity	Daily or as needed	Acceptable CCV	Correct problem and recalibrate	Analyst	WS-LC-0025

(1) Laboratory SOPs are subject to revision and updates during duration of the project, lab will use the most current revision of the SOP at the time of analysis.

Worksheets #26 & 27: Sample Handling, Custody, and Disposal

(EPA UFP-QAPP Guidance Manual, Section 3.3)

26.1 SAMPLE NUMBERING

The sample numbering system will continue to be implemented to identify each sample collected during the LTM for each site. This numbering system will ensure that each sample is uniquely labeled and will provide a tracking procedure to allow retrieval of information about each sample collected. QC samples will be numbered using the same sequential system and notes will be made in the field notebook to record which samples are QC samples; however, duplicates will not be identified to the laboratory. The sample numbering will use the AAST##### nomenclature, where AA = Area/Site Code, ST = Study ID, and ##### = 5-digit numerical code.

Table 26.1 – Sample Numbering Nomenclature

AA = AREA/SITE CODE	ST = STUDY ID	##### = 5 DIGIT NUMERICAL CODE
AL = Ash Landfill	LM = Long Term Monitoring	000## = Field QC items (e.g., Rinsate Blanks)
OB = OB Grounds	BW = Bio Wall Study	001## = Shipment QC samples (e.g., Trip Blanks)
25 = SEAD-25	SI = Site Investigation	1#### = Soil Samples
16 = SEAD-16	--	2#### = Groundwater Samples
17 = SEAD -17	--	3#### = Surface Water Samples
122 = SEAD-122E	--	4#### = Sediment Samples
26 = SEAD-26	--	--

Every sample number will be preceded by the site name designation to identify the site from which the sample was collected. The numerical component for each sample will building upon past LTM events. For database consistency, the next event sample sequence will begin with a sample ID that is one increment higher than the last sample from the previous LTM event. Sample name/numbering examples are shown in **Table 26.2**, and the complete sample list for the next round of sampling for each site is detailed on **Worksheet #18**.

Table 26.2 – Sample Name/Numbering System by Site

SITE	SITE NAME DESIGNATION	EXAMPLE SAMPLE ID (1)
OB Grounds	OBLM	OBLM0071
SEAD 25	25LM 25SI	25LM20125 25SI20001
Ash Landfill	ALBW	ALBW20375
SEAD16/17	16LM	16LM20056
	17LM	17LM20040
SEAD-122E	122SI	122SI20001
SEAD-26	26SI	26SI20001

(1) Sample numbering will begin one increment higher than the last sample in the previous LTM event (Worksheet #18).

26.2 SAMPLE HANDLING

To ensure sample authenticity and data defensibility, proper sample handing system procedures will be followed from the time of sample collection to final sample disposal. The Contractor Sample Team Lead or designee is responsible for completing the sample bottle label and chain of Custody CoC form, sample collection, sample packing, and coordination of sample shipment. The PFAS samples will be sent for analytical testing to TestAmerica Laboratories in West

Sacramento, California via FedEx or UPS Next Day Delivery service. All other samples will be sent to the analytical laboratory, Katahdin in Scarborough, Maine via FedEx or UPS Next Day Delivery service.

The laboratory receiving staff and/or custodians will acknowledge the sample receipts upon arrival. The laboratory analytical technicians will prepare and analyze the field samples in accordance with the analytical SOPs. The field samples and all extracts will be stored at the laboratory for 30 days after a final report has been submitted to Parsons. The laboratory hazardous waste manager will be responsible for the final sample disposal upon notice from the Contractor Project Chemist.

Table 26.2 – Responsibilities for Sample Handling, Custody, and Disposal

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT	
Sample Collection (Personnel/Organization)	Parsons Sample Team Lead or designee
Sample Packaging (Personnel/Organization)	Parsons Sample Team Lead or designee
Coordination of Shipment (Personnel/Organization)	Parsons Project Chemist
Type of Shipment/Carrier	FedEx or UPS Next Day Delivery
SAMPLE RECEIPT AND ANALYSIS	
Sample Receipt (Personnel/Organization)	Sample receiving supervisor, Katahdin/TestAmerica-W. Sacramento
Sample Custody and Storage (Personnel/Organization)	Sample receiving supervisor, Katahdin/TestAmerica- W. Sacramento
Sample Preparation (Personnel/Organization)	Analyst, Katahdin/TestAmerica-W. Sacramento
Sample Determinative Analysis (Personnel/Organization)	Analyst, Katahdin/TestAmerica-W. Sacramento
SAMPLE ARCHIVING	
Field Sample Storage (No. of days from sample collection)	60 days
Sample Extract/Digestate Storage (No. of days from extraction/digestion)	40 days
SAMPLE DISPOSAL	
Personnel/Organization	Sample receiving supervisor, Katahdin/TestAmerica-W. Sacramento
Number of Days from Analysis	60 days, or when notified by Parsons project chemist

26.2.1 Sample Labeling

Sample labels will include, at a minimum, project name, project number, sample ID, date/time collected, analysis group or method, preservative, and sampler's name. Labels will be taped to the jar or sample bag prior to sample collection to ensure that they do not separate.

26.3 FIELD SAMPLE CUSTODY PROCEDURES (SAMPLE COLLECTION, PACKAGING, SHIPMENT, AND DELIVERY TO LABORATORY)

Samples will be collected by field team members under the supervision of the Contractor Sample Team Lead. The sampling team will document the sample collection in a field log book. Samples will be cushioned if necessary with packaging material and placed into coolers along with the CoC. Coolers will be shipped to the laboratory via next day delivery, with the bill number indicated on the CoC (to relinquish custody). Upon delivery, the laboratory will log in each cooler and report the status of the samples.

The following addresses will be used for sample shipment of PFAS:

TestAmerica West Sacramento
880 Riverside Parkway
West Sacramento, CA 95605
Tel.: (916) 373-5600

The following address will be used for all other sample shipments:

Katahdin Analytical Services
600 Technology Way
Scarborough, ME 04074
Tel.: (207) 874-2400

26.3.1 LABORATORY SAMPLE CUSTODY PROCEDURES (RECEIPT OF SAMPLES, ARCHIVING, DISPOSAL)

All laboratory sample receipt, internal custody and sample archiving, and disposal procedures shall be completed in accordance with Katahdin SOPs: SD-902-11 and SD-903-05 and TestAmerica West Sacramento SOPs: WS-QA-0003, WS-QA-0034, and WS-EHS-0001.

26.3.2 SAMPLE IDENTIFICATION PROCEDURES

Upon opening the cooler at the analytical laboratory, the receiving clerk will sign the CoC. Then the sample containers in the cooler will be unpacked and checked against the client's CoC. Any discrepancies noted with the samples will be noted on the COC upon receipt. The clerk will deliver the CoC (and any other paperwork) to the Laboratory PM for entry into the Laboratory Information Management System (LIMS) and for client notification.

The laboratory will send sample login forms to the data validator to check sample IDs and parameters are correct. The field logbook will identify the sample ID with the location, depth, date/time collected, and the parameters requested. The laboratory will assign each field sample a laboratory sample ID based on information in the CoC.

26.3.3 CHAIN-OF-CUSTODY PROCEDURES

COC forms will include, at a minimum, laboratory contact information, client contact information, sample information, and relinquished by/received by information. Sample information will include sample ID, date/time collected, number and type of containers, preservative information, analysis method, and comments. The CoC will also have the sampler's name and signature. The CoC will link the location of the sample from the field logbook to the laboratory receipt of the sample. The laboratory will use the sample information to populate the LIMS database for each sample.

26.3.4 NON-CONFORMANCE

The Laboratory Project Managers will contact the Contractor Project Chemist to resolve any issues encountered during sample receipt and login. The Contractor Project Chemist will coordinate with the Contractor Sample Team Lead and other personnel as necessary to resolve the issues.

Worksheet #28: Analytical Quality Control and Corrective Action

(EPA UFP-QAPP Guidance Manual, Section 3.4 and Tables 4, 5, and 6)

The tables in this worksheet describe the requirements for laboratory analysis of QC samples (e.g., laboratory control samples, method blanks, matrix spikes, etc.) for each analytical method used. The tables below detail the QC sample frequency, method/SOP QC acceptance criteria, corrective actions to be taken in the event analyses do not meet the acceptance criteria and the person(s) responsible for implementing corrective actions, and measurement performance criteria.

28.1 VOCS BY EPA SW-846 METHOD 8260C IN GROUNDWATER

Matrix: Groundwater
Analytical Group: VOCs
Analytical Method: EPA SW-846 Method 8260C
SOP: CA-202

Table 28.1a - Quality Control and Corrective Actions for Analysis of VOCs in Groundwater

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT-SPECIFIC MPC
Method Blank	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Surrogate	Four per sample: Dibromofluoromethane 1,2-Dichloroethane-d4 Toluene-d8 4-Bromofluorobenzene (BFB)	QSM Appendix C limits as listed in the table below.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparation batch of twenty or fewer samples of similar matrix.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT-SPECIFIC MPC
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	As specified on the chain-of-custody by Parsons	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD of all analytes ≤ 20% (between MS and MSD	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Internal Standard (IS)	Four per sample: Pentafluorobenzene Chlorobenzene-d5 1,4-dichlorobenzene-d4 1,4-difluorobenzene	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	Not applicable (NA)	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). The parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.	Parsons Data Validator or Project Chemist	See Worksheet #12
Equipment Blank (EB)	1 per week for SEAD 25 and Ash Landfill sites. NA for all other sites	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12
Trip Blank (TB)	1 per cooler	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12.

Table 28.1b - LCS/MS/MSD Control Limits for VOCs in Groundwater

COMPOUNDS	LCS/MS/MSD CONTROL LIMITS (%R)
1,1,1-Trichloroethane	74-131
1,1,2,2-Tetrachloroethane	71-121
1,1,2-Trichloro-1,2,2-Trifluoroethane	70-136
1,1,2-Trichloroethane	80-119
1,1-Dichloroethane	77-125
1,1-Dichloroethene	71-131
1,2,4-Trichlorobenzene	69-130
1,2-Dibromo-3-chloropropane	62-128
1,2-Dibromoethane	77-121
1,2-Dichlorobenzene	80-119
1,2-Dichloroethane	73-128
1,2-Dichloropropane	78-122
1,3-Dichlorobenzene	80-119
1,4-Dichlorobenzene	79-118
Acetone	39-160
Benzene	79-120
Bromodichloromethane	79-125
Bromoform	66-130
Carbon disulfide	64-133
Carbon tetrachloride	72-136
Chlorobenzene	82-118
Chlorodibromomethane	74-126
Chloroethane	60-138
Chloroform	79-124
Cis-1,2-Dichloroethene	78-123
Cis-1,3-Dichloropropene	75-124
Cyclohexane	71-130
Dichlorodifluoromethane	32-152
Ethyl benzene	79-121
Isopropylbenzene	72-131
Methyl Acetate	56-136

COMPOUNDS	LCS/MS/MSD CONTROL LIMITS (%R)
Methyl bromide	53-141
Methyl butyl ketone (2-Hexanone)	57-139
Methyl chloride	50-139
Methyl cyclohexane	72-132
Methyl ethyl ketone (2-Butanone)	56-143
Methyl isobutyl ketone (4-Methyl-2-pentanone)	67-130
Methyl Tert-butyl Ether	71-124
Methylene chloride	74-124
Styrene	78-123
Tetrachloroethene	74-129
Toluene	80-121
Total Xylenes	79-121
Trans-1,2-Dichloroethene	75-124
Trans-1,3-Dichloropropene	73-127
Trichloroethene	79-123
Trichlorofluoromethane	65-141
Vinyl chloride	58-137
1,2-Dichloroethane-d4 (Surrogate)	81-118
4-Bromofluorobenzene (Surrogate)	85-114
Dibromofluoromethane (Surrogate)	80-119
Toluene-d8 (Surrogate)	89-112

28.2 MEE IN GROUNDWATER BY RSK-175

Matrix: Groundwater
Analytical Group: MEE
Analytical Method: RSK-175
SOP: CA-336

Table 28.2 - Quality Control and Corrective Actions for Analysis of MEE in Groundwater

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Method Blank	One per preparation batch of 20 or fewer samples.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Department Manager	Same as Method/SOP QC Acceptance Limits.
LCS	One per batch of up to 20 samples.	The laboratory must use the QSM Appendix C Limits for batch control.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Department Manager	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One MS per 10 field samples	A laboratory must use the QSM Appendix C Limits for batch control. RPD of all analytes = 20% (between MS and MSD	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Department Manager	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). The parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.	Parsons Data Validator or Project Chemist	See Worksheet #12
Equipment Blank (EB)	1 per week for SEAD 25 and Ash Landfill sites. NA for all other sites	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12

28.3 NITRATE AND NITRITE IN GROUNDWATER BY EPA METHOD 353.2

Matrix: Groundwater
Analytical Group: Nitrate and Nitrite
Analytical Method: EPA Method 353.2
SOP: CA-728

Table 28.3 - Quality Control and Corrective Actions for Analysis of Nitrate and Nitrite in Groundwater

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 90-110	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MS	One set for every set 20 samples	%R must be within: 90-110	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ. Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	Correct problem and reanalyze sample and duplicate.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). The parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.	Parsons Data Validator or Project Chemist	See Worksheet #12

Equipment Blank (EB)	1 per week for SEAD 25 and Ash Landfill sites. NA for all other sites	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12

28.4 SULFATE AND CHLORIDE IN GROUNDWATER BY EPA METHOD 300.0

Matrix: Groundwater
Analytical Group: Chloride and Sulfate
Analytical Method: EPA Method 300.0
SOP: CA-742

Table 28.4 - Quality Control and Corrective Actions for Analysis of Chloride and Sulfate in Groundwater

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 90-110	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MS	One set for every set 20 samples	%R must be within: 90-110	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ. Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	Correct problem and reanalyze sample and duplicate.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). The parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.	Parsons Data Validator or Project Chemist	See Worksheet #12

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Equipment Blank (EB)	1 per week for SEAD 25 and Ash Landfill sites. NA for all other sites	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12

28.5 COPPER, LEAD, IRON, AND SODIUM IN GROUNDWATER BY EPA SW-846 METHOD 6010C

Matrix: Groundwater
Analytical Group: Metals
Analytical Method: EPA SW-846 Method 6010C
SOP: CA-608

Table 28.5a - Quality Control and Corrective Actions for Analysis of Copper, Lead, Iron, and Sodium in Groundwater

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control.	Perform PDS and SD. Only qualify the parent sample of the MS/MSD if the PDS or SD (whichever is applicable) fail criteria. Discuss failures in the case narrative.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD)	Perform PDS and SD. Only qualify the parent sample of the MS/MSD if the PDS or SD (whichever is applicable) fail criteria. Discuss failures in the case narrative.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Post-digestion Spike	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible)	Recovery within 80-120%	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution. Qualify the parent sample and discuss in the case narrative.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution	One per preparatory batch if MS or MSD fails	Five-fold dilution must agree within ± 10% of the original measurement.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution. Qualify the parent sample and discuss in the case narrative.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed).	Parsons Data Validator or Project Chemist	See Worksheet #12

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
			The parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.		
Equipment Blank (EB)	1 per week for SEAD 25 site. NA for all other sites	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12

Notes: Method of standard addition is not required in this project.

Table 28.5b – LCS/MS/MSD Control Limits for Analysis of Copper, Lead, Iron, and Sodium in Groundwater

METALS	LCS/MS/MSD CONTROL LIMITS (%R)
Copper	86-114
Iron	87-115
Lead	86-113
Sodium	87-115

28.6 TAL METALS, EXCEPT MERCURY, BY EPA SW-846 METHOD 6020A

Matrix: Groundwater
Analytical Group: Metals
Analytical Method: EPA SW-846 Method 6020A
SOP: CA-627

Table 28.6a - Quality Control and Corrective Actions for Analysis of TAL Metals (Except Mercury) in Groundwater

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Internal Standard (IS)	Every field sample, standard and QC sample	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5- fold dilutions until criteria is met. For failed QC samples, correct problem, and rerun all associated failed field sample	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits
MS	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control.	Perform PDS and SD. Only qualify the parent sample of the MS/MSD if the PDS or SD (whichever is applicable) fail criteria. Discuss failures in the case narrative	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MSD	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control. MSD or MD: RPD of all analytes = 20% (between MS and MSD).	Perform PDS and SD. Only qualify the parent sample of the MS/MSD if the PDS or SD (whichever is applicable) fail criteria. Discuss failures in the case narrative.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Post-digestion Spike	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution. Qualify the parent sample and discuss in the case narrative.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
ICP Serial Dilution (not applicable for rinsate blanks)	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution. Qualify the parent sample and discuss in the case narrative.	Analyst, Laboratory Department	Same as Method/SOP QC Acceptance Limits.

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
				Manager, and Data Validator	
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). The parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.	Parsons Data Validator or Project Chemist	See Worksheet #12
Equipment Blank (EB)	NA	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12

Notes: Method of standard addition is not required in this project.

Table 28.6b – LCS/MS/MSD Control Limits for Analysis of TAL Metals (Except Mercury) in Groundwater

METALS	LCS/MS/MSD CONTROL LIMITS (%R)
Aluminum	84-117
Antimony	85-117
Arsenic	84-116
Barium	86-114
Beryllium	83-121
Cadmium	87-115
Calcium	87-118
Chromium	85-116
Cobalt	86-115
Copper	85-118
Iron	87-118
Potassium	87-115
Magnesium	83-118
Manganese	87-115
Nickel	85-117
Lead	88-115
Selenium	80-120
Silver	85-116
Sodium	85-117
Thallium	82-116
Vanadium	86-115
Zinc	83-119

28.7 TOC IN GROUNDWATER BY EPA SW-846 METHOD 9060A

Matrix: Groundwater
Analytical Group: TOC
Analytical Method: EPA SW-846 Method 9060A
SOP: CA-763

Table 28.7 - Quality Control and Corrective Actions for Analysis of TOC in Groundwater

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Method Blank	One per analytical batch of 20 or fewer samples.	No target analytes > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Same as Method/SOP QC Acceptance Limits.
LCS	One per analytical batch of 20 or fewer samples.	%R must be within 80-120	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MS	One for every set 10 samples	%R must be within: 75-125	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per 20 samples.	RPD ≤20 for samples >3X the LOQ, <100% RPD for samples <3X the LOQ.	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). The parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.	Parsons Data Validator or Project Chemist	See Worksheet #12

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Equipment Blank (EB)	1 per week Ash Landfill site. NA for all other sites	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12

28.8 MERCURY IN GROUNDWATER BY EPA SW-846 METHOD 7470A

Matrix: Groundwater
Analytical Group: Mercury
Analytical Method: EPA SW-846 Method 7470A
SOP: CA-615

Table 28.8 - Quality Control and Corrective Actions for Analysis of Mercury in Groundwater

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch.	82-119%R per the DoD QSM.	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MS	One per preparatory batch.	82-119%R per the DoD QSM.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
MSD	One per preparatory batch.	82-119%R per the DoD QSM. MSD or MD: RPD of all analytes = 20% (between MS and MSD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). The parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.	Parsons Data Validator or Project Chemist	See Worksheet #12
Equipment Blank (EB)	NA	See Worksheet #12	NA for laboratory	Parsons Data Validator or Project Chemist	See Worksheet #12

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
			Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.		

28.9 PFAS IN GROUNDWATER BY EPA METHOD 537 MODIFIED

Matrix: Groundwater
Analytical Group: PFAS
Analytical Method: EPA Method 537 Modified
SOP: WS-LC-0025

Table 28.9 - Quality Control and Corrective Actions for Analysis of PFAS in Groundwater

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
Internal Standards (Isotope Dilution Analytes, spiked prior to extraction)	Every sample, spiked sample, standard, and method blank	% recovery for each IS in the original sample (prior to dilutions) must be within 25-150%	Reanalyze once. Assess matrix, dilute and/or re-extract as needed. Evaluate impact on data.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch.	QSM or laboratory statistically derived control limits (70-130 until limits are established).	Reanalyze LCS once. If acceptable, report. Evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
MS	One per preparatory batch.	QSM or laboratory statistically derived control limits (70-130 until limits are established)	Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair if laboratory error is indicated.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
MSD	One per preparatory batch.	QSM or laboratory statistically derived control limits (70-130 until limits are established), RPD ≤ 30%.	Evaluate the data, and re-prepare/reanalyze the native sample and MS/MSD pair if laboratory error is indicated.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). The parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.	Parsons Data Validator or Project Chemist	See Worksheet #12
Equipment Blank (EB)	1 per day from groundwater sampling equipment 1 per day from drilling equipment	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12
Field Blank (FB)	1 per day	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12
Trip Blank (TB)	1 per cooler that includes PFAS samples	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12.

Worksheet #29: Project Documents and Records

(EPA UFP-QAPP Guidance Manual, Section 3.5.1)

29.1 PROJECT DOCUMENT AND RECORDS

All final document files, including reports, figures, and tables will be submitted in electronic format (both Microsoft Office 2007 or later and portable document format (.pdf)) on compact disk (CD)-read-only format (ROM). The tables below list the project documents and records associated with the groundwater sampling to support the LTM.

Table 29.1 – Sample Collection and Field Records(1)

DOCUMENT/RECORD	GENERATION	VERIFICATION	STORAGE LOCATION/ARCHIVAL
Field logbook or data collection sheets	Field Engineer, Parsons	Field Team Member, Parsons	Project File/Parsons-Boston Office
Chain-of-Custody Forms	Sampler, Parsons	Field Team Lead, Parsons	Project File/Parsons-Boston Office
Bills	Sampler, Parsons	Field Team Lead, Parsons	Project File/Parsons-Boston Office
Contractor Daily QC Reports	Field Engineer, Parsons	Field Team Member, Parsons	Project File/Parsons-Boston Office
Deviations	Field Engineer, Parsons	Field Team Member, Parsons	Project File/Parsons-Boston Office
Corrective Action Reports	Field Engineer, Parsons	Field Team Member, Parsons	Project File/Parsons-Boston Office
Correspondence	Various Project Team Members	Various Project Team Members	Project File/Parsons-Boston Office

Table 29.2 – Project Assessments(1)

DOCUMENT/RECORD	GENERATION	VERIFICATION	STORAGE LOCATION/ARCHIVAL
Data Validation Report	Data Validator, Parsons	Project Chemist, Parsons	Project File/Parsons-Austin Office
Data Usability Assessment Report	Data Validator, Parsons	Project Chemist, Parsons	Project File/Parsons-Austin Office

Table 29.3 – Laboratory Records (Katahdin)[2]

DOCUMENT/RECORD	GENERATION	VERIFICATION	STORAGE LOCATION/ARCHIVAL
Sample Log-in	Sample Management Technician	Log-In Technician	Digitized image, stored on local area network
Instrument Print-Out and Raw Data	Bench Analyst	Section Supervisor	Digitized image, stored on local area network
Review Checklists (Analyst)	Bench Analyst	Section Supervisor	Digitized image, stored on local area network
Review Checklists (Section Supervisor)	Section Supervisor	10-15% of data by QA staff	Digitized image, stored on local area network
PM Review Checklists	Log-in supervisor	Project Manager	Archived with project/ sampling event folder
Sample Log-in	Sample Management Technician	Log-In Technician	Digitized image, stored on local area network
Correspondence	Various Project Team Members	Various Project Team Members	Project File/Parsons-Boston Office

Table 29.4 – Laboratory Records (TestAmerica- W. Sacramento)[2]

DOCUMENT/RECORD	GENERATION	VERIFICATION	STORAGE LOCATION/ARCHIVAL
Sample Log-in	Sample Management Technician	Log-In Technician	Digitized image, stored in LIMS
Instrument Print-Out and Raw Data	Bench Analyst	Section Supervisor or designee	Digitized image, stored in LIMS
Review Checklists (Analyst)	Bench Analyst	Section Supervisor or designee	Digitized image, stored in LIMS
Review Checklists (Section Supervisor)	Section Supervisor or designee	At least 10% of data by QA staff or designee	Digitized image, stored on centralized servers
Correspondence	Various Project Team Members	Various Project Team Members	Project File/Parsons-Boston Office

[1] All project documents will be retained for 7 years. Project documents will either be stored on site at the Boston or Austin office or on the secure server until project closeout and then the documents will be moved to an off-site storage location.

[2] All project documents will be retained and archived by the laboratories for a minimum of 7 years before disposal

Worksheets #31, 32, & 33: Assessments and Corrective Action

(EPA UFP-QAPP Guidance Manual, Section 4.1.1)

These tables provide information on the required periodic assessments that will be performed during the course of the project to ensure the planned project activities are implemented in accordance with this UFP-QAPP. The type, frequency, and responsible parties of planned assessment activities to be performed for the project are summarized in the table below.

ASSESSMENT TYPE	RESPONSIBLE PARTY & ORGANIZATION	NUMBER/FREQUENCY	ESTIMATED DATES	ASSESSMENT DELIVERABLE	DELIVERABLE DUE DATE
Non-Conformance Report from the field[1]	Parsons	When there is an issue	Within 24 hours from the occurrence	Non-Conformance Report	Will submit to USACE within 30 days from the occurrence
Field Record Verification	Parsons	At the end of each sampling day	Each sampling day	At the end of each sampling event	Field Record Verification
Corrective Action Report (CAR)	Field Related: Parsons PM Lab Related: Lab's QA Manager	When occur	Within 48 hours from the occurrence	CAR	Will submit to USACE within 30 days from the occurrence
Approval of the Proposed Corrective Action	Field Related: Parsons PM Lab Related: Lab Director	When occur	Within 48 hours from the completion of the issuing of the CAR	CAR with approver's signature	Will submit to USACE within 30 days from the occurrence
Implementation of Corrective Action	Field Related: Parsons PM Lab Related: Lab Director	When occur	Immediately after the approval of the CAR	Same as above	Will submit to USACE within 30 days from the occurrence
Verification of the Corrective Action	Field Related: Parsons Project QC Manager Lab Related: Lab's QA Manager	When occur	30 days from the approval of the CAR	Completed CAR	Will submit to USACE within 30 days from the occurrence
Laboratory Analysis Data Validation	Parsons data validator	Each data package	Labs will submit data package on before 21 calendar days from sample receiving day. Parsons will complete data validation 14 calendar days from data package receiving date.	Data validation report	Will submit to USACE.

(1) The field program has been operating for many years using the same field procedures as outlined in the QAPP. Field events are short in duration. No field audits are proposed.

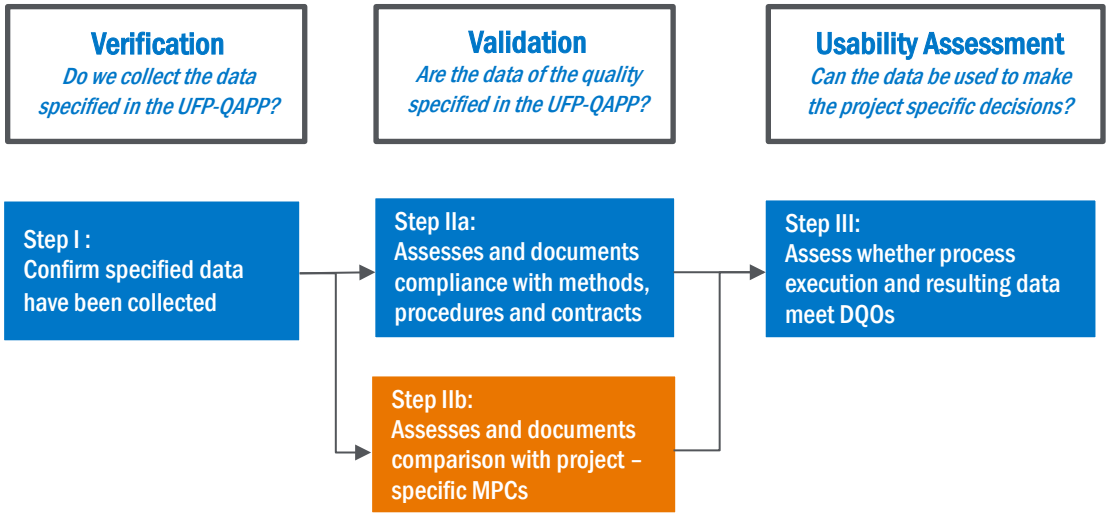
ASSESSMENT TYPE	RESPONSIBILITY FOR RESPONDING TO ASSESSMENT FINDINGS	ASSESSMENT RESPONSE DOCUMENTATION	TIMEFRAME FOR RESPONSE	RESPONSIBILITY FOR IMPLEMENTING CORRECTIVE ACTION	RESPONSIBLE FOR MONITORING CORRECTIVE ACTION IMPLEMENTATION
Non-Conformance Report from the field[1]	Parsons PM	Internal Memo	Within 24 hours from the occurrence	As directed by PM	Parsons PM and Field Engineer
Field Record Verification	Parsons Field Engineer Parsons PM	Internal Memo	Each sampling day	As directed by PM	Parsons PM and Field Engineer
Corrective Action Report (CAR)	Field Related: Parsons PM Lab Related: Lab's QA Manager	Corrective action reports (if the error is severe), updated case narratives, and corrected data submissions	Within 48 hours from the occurrence	Field Related: As directed by PM Lab Related: As directed by Lab Director	Parsons PM and Lab Director
Approval of the Proposed Corrective Action	Field Related: Parsons PM Lab Related: Lab Director	Internal Memo	Within 48 hours from the completion of the issuing of the CAR	Field Related: Parsons PM Lab Related: Lab Director	Parsons PM and Lab Director
Implementation of Corrective Action	Field Related: Parsons PM Lab Related: Lab Director	Responses to comments and report revisions	Immediately after the approval of the CAR	Field Related: Parsons Field Engineer Lab Related: Lab Technical Director	Parsons Field Engineer and Lab Technical Director
Verification of the Corrective Action	Field Related: Parsons Project QC Manager Lab Related: Lab's QA Manager	Internal Memo	30 days from the approval of the CAR	Field Related: Parsons PM Lab Related: Lab Director and Lab's QA Manager	Parsons PM and Lab Director
Laboratory Analysis Data Validation	Parsons data validator	Internal Memo	Labs will submit data package on before 21 calendar days from sample receiving day. Parsons will complete data validation 14 calendar days from data package receiving date.	Parsons Data Validator	Parsons Data Validator and Parsons PM

Worksheet #34: Data Verification & Validation Inputs

(EPA UFP-QAPP Guidance Manual, Section 5.2.1)

This worksheet lists the inputs that will be used during data verification and validation. Inputs include planning documents, field records, and laboratory records. Data verification is a check that all specified activities involved in collecting and analyzing samples have been completed and documented and that the necessary records (objective evidence) are available to proceed to data validation. Data validation is the evaluation of conformance to stated requirements, including those in the contract, methods, SOPs and the UFP-QAPP. Data validation includes evaluation of the data against the project –specific MPCs (**Worksheet #12**). Data verification and validation procedures and responsibilities are described on Worksheet #35 and Worksheet #36, respectively. Once verification and validation have been completed, a usability assessment is conducted to evaluate whether process execution and resulting data meet DQOs. Usability assessment procedures are described on Worksheet #37. The data verification, validation, and usability assessment process is summarized in **Figure 34-1**.

FIGURE 34-1 - DATA VERIFICATION, VALIDATION, AND USABILITY ASSESSMENT PROCESS



34.1 VERIFICATION AND VALIDATION INPUTS

DESCRIPTION	VERIFICATION (COMPLETENESS)	VALIDATION (CONFORMANCE TO SPECIFICATIONS)
Planning Documents/Records		
Approved QAPP	X	
Contract	X	
Field SOPs	X	
Laboratory SOPs	X	
Field Records		
Field logbooks	X	X
Equipment calibration records	X	X
Chain-of-Custody Forms	X	X
Relevant Correspondence	X	X
Change orders/deviations, when applicable	X	X
Field corrective action reports, when applicable	X	X
Analytical Data Package		
Cover sheet (laboratory identifying information)	X	X
Case narrative	X	X
Internal laboratory chain-of-custody	X	X
Sample receipt records	X	X
Sample chronology (i.e. dates and times of receipt, preparation, & analysis)	X	X
Communication records	X	X
DL/LOD/LOQ establishment and verification	X	X
Standards Traceability	X	X
Instrument calibration records	X	X
Definition of laboratory qualifiers	X	X
Results reporting forms	X	X
QC sample results	X	X
Corrective action reports, when applicable	X	X
Raw data	X	X
Electronic data deliverable	X	X

Worksheet #35: Data Verification Procedures

(EPA UFP-QAPP Guidance Manual, Section 5.2.2)

"Verification" is a completeness check that is performed before the data review process is conducted to determine whether the required information is available for validation. It involves a review of all data inputs to ensure that they are present. This step of the data review process answers whether or not the required data inputs are present. The following table summarizes the methods for data verification.

RECORDS REVIEWED	REQUIREMENT DOCUMENTS	PROCESS DESCRIPTION	RESPONSIBLE PERSON, ORGANIZATION
Field logbook	UFP-QAPP, WP, SOPs	Verify that records are present and complete for each day of field activities. Verify that all planned samples including field QC samples were collected and that sample IDs are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that any required field measurement was performed and results are documented.	Daily – Parsons Field Team Lead At conclusion of field activities – Parsons Project Manager
Chain-of-custody forms	UFP-QAPP, WP, SOPs	Verify the completeness of chain-of-custody records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (e.g., MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors.	Daily – Parsons sampler At conclusion of field activities – Parsons Project Chemist
Laboratory Deliverable	UFP-QAPP, WP, SOPs	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the CoCs to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	Before release – Lab Project Manager, Katahdin/TestAmerica-W. Sacramento Upon receipt – Parsons data validator
Corrective Action Reports	UFP-QAPP, WP, SOPs	For any non-compliance noted, verify that corrective action was implemented according to plan.	Parsons Project Chemist

Worksheet #36: Data Validation Procedures

(EPA UFP-QAPP Guidance Manual, Section 5.2.2)

"Validation" is performed to identify and qualify data that do not meet the MPCs specified on **Worksheet #12**. Data requiring validation are summarized on **Worksheet #34**. The information in these tables shows what data inputs are required for data validation as well as the processes used to conduct the validation.

36.1 VALIDATION PROCESS

General procedures for chemistry data review and management are described in SOP CHEM-01, Chemistry Data Management (**Appendix B**). Project specific elements for data validation on this project are summarized in **Tables 36.1** and **36.2** below.

Table 36.1 - Overview of Analytical Data Validation

DATA VALIDATORS: PARSONS	
Analytical Group/Method:	All Chemical Analyses
Data deliverable requirements:	Level IV data packages and EDDs
Analytical specifications:	Per UFP-QAPP, DoD QSM version 5.0, and Katahdin SOPs
Measurement performance criteria:	Per UFP-QAPP and DoD QSM version 5.0
Percent of data packages to be validated:	100%- Level IV data validation as described in SOP CHEM-01
Percent of raw data reviewed:	100%
Percent of results to be recalculated:	10%
Validation procedure:	Per UFP-QAPP, DoD QSM version 5.0 (specific to PFAS, Appendix B, Table 15)
Data validation codes:	See table below
Electronic validation program/version:	CSV file

Table 36.2 - Data Validation Codes and Definitions

DATA VALIDATION CODES	DEFINITIONS
U	Analyte was not detected and is reported as less than the Limit of Detection (LOD). The LOD has been adjusted for any dilution or concentration of the sample.
B	Blank contamination. The recorded result (<5x lab non-common contaminants or <10x lab common contaminants) is associated with a contaminated blank.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
UJ	The analyte was not detected above the reported sample LOQ. However, the reported LOQ is approximate and may or may not represent the actual LOQ necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Note: Labs will report all concentration down to Detection Limit (DL) and flag any results between DL and LOQ with "J". All non-detected will be reported as <LOD, per DoD QSM version 5.0.

Electronic data received by the laboratory is reviewed against the hard-copy data report. The automated process will include data flagging for issues related to method blanks, equipment blanks, trip blanks, ambient blanks, LCSs, MS/MSD samples, field duplicates, field triplicates, surrogate recoveries, holding time, and reconciliation of dilutions and re-extractions. All of the elements of QC, their limits, and logic for applying flags are incorporated in the ADR computer application. The software will apply data flags, as well as the reason for each flag. A final flag is applied to the data by the data validator/chemist within the ADR software after reviewing hard copy reports, evaluating all flags applied by the

software, and then selecting the most conservative flagging. Final validated ADR formatted text data files which contain all validation flags and reasons will be exported from the ADR software and then electronically imported into a Microsoft Access database. All data summary tables presented in final reports will be prepared using the database. The validated database will be made available to the client.

Worksheet #37: Usability Assessment

(EPA UFP-QAPP Guidance Manual, Section 5.2.3)

This worksheet documents procedures that will be used to perform the data usability assessment. The data usability assessment is performed at the conclusion of data collection activities, using the outputs from data verification and data validation. It is the data interpretation phase, which involves a qualitative and quantitative evaluation of environmental data to determine if the project data are of the right type, quality, and quantity to support the decisions that need to be made. It involves a retrospective evaluation of the systematic planning process, and, like the systematic planning process, involves participation by key members of the project team. The data usability assessment evaluates whether underlying assumptions used during systematic planning are supported, sources of uncertainty have been accounted for and are acceptable, data are representative of the population of interest, and the results can be used as intended, with the acceptable level of confidence.

37.1 USABILITY ASSESSMENT

37.1.1 SUMMARY OF USABILITY ASSESSMENT PROCESSES

The first step of the data usability assessment is to review the sampling design and data collection documentation for consistency and completeness with the project objectives observing any potential discrepancies. Data Validation will be the second step of the usability assessment. See **Worksheet #28** for data quality indicators associated with the analytical measurements to be used on the project. The statistical analysis step will not be performed for this project because there are not enough historical data to perform this step in the data usability assessment; however, the available data for this project will be reviewed for any indication of trends for project compounds of concern. The last step in the assessment process is to determine if the data can be used as intended. All data qualifiers will be evaluated and any possible impact to the overall data quality will be discussed in the data usability assessment report. Any data gap due to the field and/or lab error will be pointed out in the report and possible impact to the project will be discussed. Data validation will be the first step of the usability assessment. See **Worksheet #28** for data quality indicators associated with the analytical measurements to be used on the project. All data qualifiers will be evaluation and any possible impact to the overall data quality will be discussed in the data usability assessment report. Any data gap due to the field and/or lab error will be pointed out in the report.

37.1.2 DOCUMENTATION GENERATED

A data validation report will be created for each sample delivery group (SDG), including a summary of all QA/QC results associated with the SDG to provide documentation whether data generated were in control throughout sample analysis. Topics of discussion include all accuracy and precision exceedances as well as the extent of the exceedance and the acceptance criteria for Accuracy/Biased Contamination, Precision of all laboratory and field QA/QC results. The field samples affected by the exceedance and the qualifiers applied to the samples will also be documented. Field duplicate Discussion of, Sensitivity, Representativeness, and Completeness will also be included in the report. Criteria listed in the **Worksheet #12** will be examined to determine if the Measurement Performance Criteria were met. Any lab trending in the QC samples, such as high biased lab control sample for a particular analyte will also be discussed. Data summary tables will be generated in order for data reviewer to review the results in an organized manner. Footnotes will include all flag definitions.

An overall data usability report will describe the rationale for the data used and present any data limitations. The report will include a discussion of the accuracy, precision, representativeness, completeness and comparability of the data set and deviations from planned procedures and analysis and the impact on the project objectives. Maps will be generated with validated data, and will be presented in the respective annual or letter reports for each site.

37.1.3 PROCEDURES TO ASSESS PROJECT-SPECIFIC OVERALL MEASUREMENT ERROR

The Contractor will determine if quality control data is within specifications through the data validation process (**Worksheet #36**).

37.1.4 PERSONNEL RESPONSIBLE FOR PERFORMING USABILITY ASSESSMENT

The following personnel are responsible for performing usability assessments:

- Contractor Project Manager
- Contractor Project Chemist

37.1.5 IMPACTS OF QUALIFIED DATA AND PLAN DEVIATIONS

The Contractor will use all data not rejected during validation to determine the nature and extent of contamination, and to support the risk assessment. The Contractor will work with the Army and project regulators if there is a concern about the statistical validity of the sample results or to determine if sample locations with rejected data need to be re-sampled.

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Appendices

Appendix A

Contractor SOPs

Procedure # CHEM-01	Title: CHEMISTRY DATA REVIEW AND MANAGEMENT	Revision # 00
Effective Date: 05/20/2015	**Approved By:** Tammy Chang	**Last Reviewed/Revised:** 05/20/2015

1. PURPOSE

The purpose of this SOP is to describe the general procedures involved in chemistry data review and management for environmental projects. The elements involved include data verification, data validation, data usability assessment, and documentation, flagging conventions, electronic data deliverables, and data archiving.

2. RESPONSIBILITIES

Role	SOP-specific Responsibilities
Project Chemist	Ensures laboratory analytical data are managed, reviewed, validated, reported, and stored in accordance with approved requirements.
Data Manager	Ensures laboratory analytical electronic data are managed, reviewed, validated and reported in accordance with approved requirements and that data integrity is maintained.

3. RELEVANT DEFINITIONS

Term	Definition
Data Verification	The first step in the data review process. Data verification involves a completeness check to determine whether the analytical laboratory has provided the required information to permit adequate data validation and review.
Data Validation	The second step in the data review process. Data validation extends data verification and is the systematic process of evaluating whether data comply with pre-defined, project-specific requirements and criteria.
Data Usability Assessment	The third step in the data review process. The usability assessment is an evaluation based on the findings of the data verification and validation steps. It includes discussion of the final data flags applied to the sample results and assessment of whether the data meet project method and data quality objectives.
Data flags	Project-specific notations applied to individual analytical results to provide the data user with a qualitative assessment of the data (e.g., "estimated" or "rejected"). Data flags are also sometimes called “qualifiers.”

4. REQUIRED EQUIPMENT

Equipment	Brief Description of Function and Purpose
None	Not applicable.

Procedure # CHEM-01	Title: CHEMISTRY DATA REVIEW AND MANAGEMENT	Revision # 00
Effective Date: 05/20/2015	**Approved By:** Tammy Chang	**Last Reviewed/Revised:** 05/20/2015

5. PROCEDURE

5.1. Overview

5.1.1 The Project Chemist shall ensure that all data generated by the analytical laboratory is reviewed and managed in accordance with the project-specific work plan and/or Quality Assurance Project Plan (QAPP). Data verification, data validation, and usability assessment are the three steps of the data review process by which data adequacy and quality are examined and evaluated. After the data verification and validation steps have been performed, a data usability assessment can be completed. The usability assessment includes the discussion of the results based on the verification and validation, as well as discussion of how final data qualifiers, also known as data flags, are properly applied to the sample results. The final data flags as they were described in the usability assessment are applied to the electronic data results in the project database. The following sections of this SOP address these steps.

5.1.2 Note that not all data may require verification or validation for a given project. For example, for projects with large quantities of data, a representative selection may be verified and validated. The rest of the data may be verified without validation. Also, waste characterization and screening data are not typically subject to validation. The quantity and/or types of data to be verified and validated will be specified in the project-specific work plan and/or QAPP.

5.2. Data Verification

The Project Chemist or designee shall verify data packages received from the analytical laboratory as required during the project. Data verification will involve the reviewer conducting a completeness check to determine whether the analytical laboratory has provided the required information to permit adequate review and validation. The required information to be provided by the laboratory for the project is described in the project-specific work plan and/or QAPP. Data verification will be documented as specified in the project-specific work plan and/or QAPP.

5.3. Data Validation

Following verification, the Project Chemist or designee shall validate data packages received from the analytical laboratory as required during the project. Data validation is the systematic process of evaluating whether the data comply with pre-defined requirements and criteria of a specific project. There are three levels of data validation: Level 2, Level 3, and Level 4; with Level 2 being a more basic level of review and Level 4 being the most comprehensive. Each higher level of validation includes the level(s) below (i.e., Level 3 validation also includes Level 2 validation and Level 4 also includes Levels 2 and 3). The level of data validation required for the project and the project-specific validation criteria to be used are described in the project-specific work plan and/or QAPP.

5.3.1. Level 2 Validation

Level 2 validation of the laboratory analytical data package comprises a series of assessments concerning the compliance of sample receipt conditions, sample characteristics, and analytical results. **Table 1** shows the validation steps and requirements for Level 2 validation.

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TABLE 1
VALIDATION STEPS AND REQUIREMENTS FOR LEVEL 2 DATA VALIDATION

Validation Step (Review Items)	Validation requirement
Case Narrative	Verify necessary information are included and discussed as appropriate (e.g., parameters analyzed, description of all analytical and sample receipt problems, discussion of reasons for any QA/QC exceedances, and discussion regarding any occurrences that adversely impact sample integrity or data quality).
Corrective Action Reports (CARs), if applicable	Review report for completeness ensuring the cause and corrective action are identified, and the corrective action has been implemented.
Chain-of-Custody (COC) documentation	Examine traceability of the data from time of sample collection through reporting of results. Examine chain-of-custody records against QAPP requirements.
Sample condition upon receipt, and storage records	Verify required sample handling, receipt, and storage procedures were followed, and deviations were documented.
Sample chronology	Verify date and time samples were received, extracted and analyzed.
Sampling Methods and Procedures	Verify required analytical methods were performed, including preparation and cleanup when needed.
Holding Times	Ensure samples were prepared and analyzed within holding times specified in method, procedure, and contract requirements. If holding times were not met, confirm deviations were documented, appropriate notifications were made (consistent with procedural requirements), and appropriate approval to proceed was received prior to analysis.
Sample results	Confirm required target analytes are reported and data includes the original laboratory data qualifiers. Confirm requested concentration units are reported for each method. Review sample results and confirm requested reporting limits for all samples are present; verify results and limits are adjusted for dilutions and dry weight for soils where applicable. Determine which result should be used to make project decisions if multiple analyses were performed for any analyte.
QA/QC Samples	Evaluate all sample-related quality control (QC) data against designated acceptance criteria for accuracy and precision listed in the project specific work plan and/or QAPP (including method blank detections, surrogate recoveries, laboratory control sample [LCS] recoveries, duplicate precision, and matrix spike/matrix spike duplicate [MS/MSD] recoveries and precision). Verify laboratory QC is linked to sample data via batch identifiers. Verify QC samples were performed at the required frequency.

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5.3.2. Level 3 Validation

Level 3 validation of the laboratory analytical data package consists of the Level 2 validation plus an assessment of the compliance of instrument-related QC. **Table 2** shows the validation steps and requirements for Level 3 validation.

TABLE 2
VALIDATION STEPS AND REQUIREMENTS FOR LEVEL 3 DATA VALIDATION

Validation Step (Review Items)	Validation requirement
Initial instrument calibration records	Confirm compliance with project requirements and/or acceptance criteria included in the project work plan and/or QAPP.
Secondary source standard (initial calibration verification)	
Continuing calibration verification	
Calibration blanks	
Method specific instrument performance checks (e.g., tunes for mass spectrometry methods, DDT/Endrin breakdown checks for pesticides and Aroclors, instrument blanks and dilution test, post digestion spike, and interference checks for Inductively Coupled Plasma (ICP) methods)	
Sample Results	Evaluate sample results by comparing instrument-related QC data to the requirements and guidelines present in the project work plan and/or QAPP.

5.3.3. Level 4 Validation

Level 4 validation of the laboratory analytical data package consist of all items listed for Level 2 and Level 3 validation, plus the validation of the overall data set. **Table 3** shows the validation steps and requirements for Level 4 validation.

5.3.4. Flagging Conventions

The final data flags used to qualify data shall be applied by the Project Chemist during the data validation process. Final data flags applied to data are discussed in the project-specific data usability assessment, then the Data Manager uses the final data flag discussion from the usability assessment to apply those flags to the electronic data in the project database. The type of final data flags and their definitions are specific to the project and are listed in the project specific work plan and/or project QAPP.

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TABLE 3
VALIDATION STEPS AND REQUIREMENTS FOR LEVEL 4 DATA VALIDATION

Validation Step (Review Items)	Validation requirement
Patterns and Trends	Review project data for patterns and trends in the sample and/or QC data that may indicate systematic issues/bias across the entire data set.
Raw instrument data	Review chromatograms, ion spectra, and manual spot check of electronic calculations, including chromatograms from dual column and/or dual detectors.
Manual Integrations	Review all manual integrations to ensure they were properly performed and documented. Raw data records where manual integrations were performed must include the following: (1) chromatogram before manual integration and after manual integration, (2) notation of the cause and justification for performing the manual integration, (3) date and signature or initials of the person performing the manual integration.
Laboratory Sensitivity	Evaluate lab sensitivity by identifying whether reporting limits met those required by the project work plan and/or QAPP and that non-detect values were reported at concentrations below the required Project Action Limits listed in the project work plan and/or QAPP (as applicable). Evaluate low-level detections to identify possible false-positive results below the limit of detection but detected at or above the detection limit based on data reproducibility, blank detections, and chromatographic interference.
Standards	Determine the traceability of all chemical standards used in preparation and analysis and that they method or procedural requirements.
Deviations (if applicable)	Review any deviations from planned activities (e.g. work plan and/or QAPP deviations) and their impacts on the data usability.

5.4. Corrective Actions

If the data reviewer assesses the data package to be incomplete, in error, or otherwise requiring revision, the Project Chemist or designee shall contact the Laboratory Project Manager (PM) and ensure corrective actions are initiated as soon as possible to rectify the issue(s). If necessary, the Project Chemist shall instruct the Laboratory PM to correct and reissue the applicable data package(s). Data verification and validation shall be repeated for the revised elements of the data package(s).

5.5. Data Usability Assessment and Documentation

Following validation, the Project Chemist or designee shall conduct a usability assessment for the data packages received from the analytical laboratory as required during the project. The data usability assessment uses the results of the data verification and validation steps, including discussion of the final data flags applied to the sample results (see Section 5.3.4). The data usability assessment involves evaluating whether the data meet project method and data quality objectives. The Project Chemist or designee shall document the findings of the data usability assessment in the format specified in the project-specific work plan and/or QAPP (e.g., report, checklist, etc.).

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5.6. Electronic Data Deliverables (EDD)

5.6.1 The Data Manager shall import and store electronic analytical data in a project-specific database using electronic processing as a means to maintain and assure the accuracy, consistency, and integrity of the data. Electronic data evaluation will follow a systematic process of review using a set of logical data queries and/or validation software to ensure that electronic data comply with pre-defined project requirements. The Data Manager shall apply data flags to electronic data as determined during the validation process (see Section 5.3.4) and then the Data Manager shall use the validated data to generate data summary tables that include the validation flags. The Data Manager is responsible for verifying the data summary tables for accuracy and completeness by comparison to the validated hard copy laboratory reports.

5.6.2 The required EDD format and government database submittal deliverables are specific to the project and are described in the project-specific work plan and/or QAPP. The Data Manager is responsible for ensuring data are uploaded to required government databases in accordance with project requirements.

5.7. Data Archive

Electronic project files, such as laboratory data (reports and EDDs), validation checklists and/or validation reports, project database, and data tables shall be stored and maintained as described in the project-specific work plan and/or QAPP. Hard-copy project files shall be stored at the Parsons office where the Project Chemist is located until project closeout, at which point the documents shall be moved to the Parsons Project Manager's Office or an approved off-site storage location. All Department of Defense project related laboratory and validation documents shall be stored for minimum of seven years from the acceptance of data by the client.

6. REFERENCES

Reference Title (Author)	Brief summary of relevance to this procedure
Uniform Federal Policy for Quality Assurance Project Plans Part 1: UFP-QAPP Manual, Final Version 1 March 2005.	Defines and describes requirements for data review elements: verification, validation and data usability.

7. EXHIBITS

None.

8. REVISION HISTORY

Rev.	Date	Summary of Changes	Reason for Revision
00	05/20/15	Initial Release	n/a

Procedure # ENV-02	Title: GROUNDWATER SAMPLING	Revision # 00
Effective Date: 02/18/15	**Approved By:** Thomas Mills, PG	**Last Reviewed/Revised:** 02/18/15

1. PURPOSE

The purpose of this SOP is to describe the general methods to be employed when collecting groundwater samples for analysis during munitions response projects. Types of sampling methods include low flow techniques, Hydrasleeve™ sampler, and direct push groundwater sampling (hydropunch). Proper collection procedures are necessary to assure the quality and integrity of the samples.

2. RESPONSIBILITIES

Role	SOP-specific Responsibilities
Project Chemist	Specifies the types and quantities of soil samples to be collected. Monitors sample collection through communication with project team and field document review to confirm required samples are collected. Coordinates with analytical laboratory during sampling.
Sample Team Leader	Responsible for implementing the sampling activities outlined in the work plan. Ensures required QC and QA samples are collected. Records sample collection on field documentation.
Sample Team Assistant	Assists the Sample Team Leader with sample collection and other sampling activities. The role of Sample Team Assistant may be performed by the accompanying UXO Tech II.
UXO Tech II (or higher)	If explosive hazards are present at the sample location, acts as MEC escort and conducts anomaly avoidance prior to sample collection. May act as Sample Team Assistant.

3. RELEVANT DEFINITIONS

Term	Definition
None	Not applicable.

4. REQUIRED EQUIPMENT

Equipment	Brief Description of Function and Purpose
Sampling tools	*Low flow and direct push groundwater sampling*: Submersible or peristaltic pump, clean tubing, graduated cylinder, purge containers. *Hydrasleeve™ Method*: Hydrasleeve™ sample bags, measured line, and sampler weights.
Sample containers	Bottles as specified in the approved work plan for sample containerization. Coolers for sample shipment.
Logbook	For documentation of the sampling activities.
GPS Unit	To record coordinates of collected sample locations.
Water Level Indicator	To measure depth to static water level and total depth of well.

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Equipment	Brief Description of Function and Purpose
Water Quality Meter	To measure water quality parameters: Temperature, pH, conductivity, turbidity, DO, and ORP or as specified in the approved work plan.
Photo-ionization Detector (PID)	To measure volatile compounds at the wellhead.

5. PROCEDURE

5.1. Health and Safety

All elements of this procedure will be conducted in accordance with the approved site safety and health plan, including but not limited to specified requirements for training, personal protective equipment (PPE), exposure monitoring and air sampling, etc. The UXOSO or designated representative will review the relevant site-specific activity hazard analyses (AHAs) prior to implementing this SOP.

5.2. General Requirements for all Sample Methods

5.2.1. Documentation

The Sample Team Leader or designee shall record the description of sample locations, soil type, and any other relevant or notable details in the Field Sampling Logbook and/or on project-specific sampling forms. The Sample Team Leader or designee shall also record the sample locations using a global positioning system (GPS) unit (e.g., Trimble® GeoXT™ or similar) and document sample coordinates in the Field Sampling Logbook. The Sample Team Leader or designee shall record other information as specified in the approved work plan, including completion of a Daily Quality Control (QC) Report (DQCR) on any day that samples are collected.

5.2.2. Sampling Handling and Shipment

5.2.2.1. The Sample Team Leader is responsible for ensuring samples are packaged and shipped to the analytical laboratories in accordance with the approved work plan. Methods to be used for sample handling and shipment are described in the approved work plan. The Sample Team Leader or designee shall document sample details on the CoC form. The completed CoC form will be included with the shipped sample(s).

5.2.2.2. Sample purge water and equipment decontamination water may be required to be containerized as investigation-derived waste (IDW) and analyzed. The Sample Team Leader will review the requirements in the Waste Management Plan (included as a part of the work plan) for chemical analysis and proper disposal of IDW.

5.2.3. Field Instrument Calibration and Sample Analysis

5.2.3.1. When groundwater samples are being collected, the water quality meter and dissolved oxygen (DO) sensor will be checked at the beginning of each day. The Sample Team Leader or designee shall bump check the water quality meter to ensure the sensors are within 5 percent of the calibration standards (or as specified in the work plan) for: pH 4, pH 7, pH 10, Zobell's ORP Solution (or similar), Turbidity 0 NTUs and Conductivity Standard 1413µS. The Sample Team Leader or designee shall also bump check the DO sensor in a sodium sulfite solution to ensure the sensor is reading less than 0.1 mg/L of oxygen in the zero oxygen solution. If any parameter is outside 5 percent, that parameter will be calibrated and checked again.

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5.2.3.2. If specified in the work plan, the photo ionization detector (PID) will be used to screen the breathing zone around the open well casing. Air monitoring data shall be recorded on a field form or in the field notebook.

5.2.3.3. Collected groundwater samples shall be analyzed in the field and/or at the analytical laboratory as described in the approved work plan. The Sample Team Leader or designee shall collect the quantities and types of Quality Assurance (QA)/QC samples specified in the approved work plan to ensure proper QC review of each sampling event.

5.2.4. Anomaly Avoidance

If munitions and explosives of concern (MEC) hazards are present at the proposed sample location, a MEC Escort will practice anomaly avoidance in accordance with **SOP MEC-03, MEC Avoidance and Escort** before sample collection from non-existing wells (e.g., groundwater samples collected using Hydropunch). Once the proposed location has been cleared for subsurface anomalies, the sample can be advanced. Down-hole anomaly avoidance shall also be practiced as described in **SOP MEC-03, MEC Avoidance and Escort**. If a subsurface anomaly is detected at the planned sample location, the sample location will be moved to a nearby alternative point and the process will be repeated until a suitable sample location is found. The Sample Team Leader or designee shall record sampling locations that are moved from those proposed in the work plan in the Field Sampling Logbook, along with a brief explanation.

5.3. Sampling Methods for Groundwater

5.3.1. General Preparatory Steps for Groundwater Sampling

The following general steps shall be completed when preparing for collection of groundwater samples:

1. The Sample Team Leader shall review the applicable section(s) of the work plan to confirm the sample location, quantities, required sample containers, and other relevant information.
2. The Sample Team will navigate to the sample location, make initial observations, and complete the required documentation (see Section 5.2.1).
3. The Sample Team shall don clean gloves before each sampling event.
4. The Sample Team shall assemble the necessary sampling equipment and supplies, sample containers, decontamination materials, etc in the sampling area. If on-site decontamination is required, arrange the necessary supplies in a nearby but separate location, away from the wellhead. All equipment entering the well shall be decontaminated.
5. The Sample Team shall calibrate required equipment and document the calibration on an equipment calibration form.

5.3.2. Low Flow Techniques for Groundwater

5.3.2.1. This sampling method is designed to ensure that a representative groundwater sample is collected while minimizing the volume of purge water generated. This method dictates that pre-sample purging (the removal of standing water from a well and filter pack immediately prior to sample collection) be done at very low flow rates. Low flow purging and sampling involves the use of a submerged or peristaltic pump that can be adjusted to deliver ground water to the surface at rates from less than 100 ml per minute to a maximum of 1 liter per minute. The purpose of this technique is the recovery of representative samples of the water from the soil formation adjacent to the well screen. Stagnant water above the screen and below will not usually be purged or sampled. The technique eliminates the need for collection and costly disposal of several well volumes of groundwater as investigative derived waste (IDW) from wells containing contaminated water.

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5.3.2.2. During low flow purging and sampling the pump intake is placed within the lower depths of the screened interval and the water pumped from the well is monitored for a number of water quality parameters using a flow through cell and field instrumentation. The water level will also be monitored to ensure that draw down is kept to a minimum as specified in the approved work plan. Sampling commences when the measured parameters have stabilized and turbidity is at an acceptable and constant level. Specific procedure for conducting groundwater sampling using low flow techniques are as follows:

5.3.2.3. Preparation: The steps listed in Section 5.3.1 shall be completed prior to sample collection using low flow methods.

5.3.2.4. Groundwater Sampling: Following the preparatory actions described above, the Sample Team shall complete the following steps to collect low flow groundwater samples:

1. Open well and measure depth to static water level and total depth of the well using an electronic water level meter. Record these measurements into the project specific log or electronic form or application.
2. Lower pump slowly into the well to a depth a couple feet above the bottom of the well screen.
3. Allow water column to equilibrate then measure static water level again, use this measurement as the reference point for drawdown.
4. Begin purging the well. Using a graduated cylinder, establish the maximum flow rate that does not cause drawdown of the well (commonly a rate between 100ml and 300ml per minute) or as specified in the approved work plan.
5. If a well is pumped dry at the lowest consistent flow rate the sampler can establish, then the well is considered properly purged, and groundwater samples will be collected when 80% of the initial well water volume is recharged.
6. Connect tubing through the water quality meter, record initial water quality parameters, then continue recording readings every 3 to 5 minutes, or as specified in the approved work plan. If using an electronic form or groundwater sampling application (e.g. In-Situ low flow test) ensure all required information has been entered prior to starting the flow to the meter.
7. Monitor parameter until all have stabilized. Typical stabilization requirements are:

   (a) pH: ± 0.2 pH units
   (b) Conductivity: ± 3% of reading
   (c) Dissolved Oxygen: ± 10% or reading or ± 0.2 mg/l, whichever is greater
   (d) Eh or ORP: ± 20 mV
   (e) Turbidity: ± 10% prior reading or ± 1.0 NTU
   (f) Temperature: +/- 1ºC

8. Arrange the sample containers in the order of use. VOCs first, if required, SVOCs second, if required followed by all other samples.
9. Label each sample container with sample ID, date, time, analysis, and other information required on the sample label. Immediately place the filled containers in the coolers(s) on ice.
10. Record sample types, amounts collected, time, and date of collection in the field logbook and on the monitoring well purge and sample log (**Exhibit 1**).
11. Perform post-sampling activities (Section 5.3.5).

5.3.3. Hydrasleeve™

5.3.3.1. The HydraSleeve™ groundwater sampling device is designed to collect a representative groundwater sample from a well while eliminating the need to purge the well. The sample is collected from a specific depth within the screened interval of the well without mixing fluid from other depth

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intervals. Because the HydraSleeve™ sampler does not require purging, field measurements of groundwater parameters (e.g., temperature, pH, conductivity, etc.), normally taken during purge sampling, are not required to evaluate whether the groundwater parameters have stabilized prior to sampling. However, verify in the approved work plan that measuring and recording of water quality parameters are not required when sampling with the HydraSleeve™.

5.3.3.2. The displacement of well water caused by placement of a single HydraSleeve™ sampler is minimal (<100 milliliters). Because the sampler does not disturb the water column significantly, long equilibrations times following insertion of the sampler into the well are generally unnecessary. To obtain a groundwater sample, the HydraSleeve™ is pulled upward on the suspension line through the zone of interest, which causes water to enter the one-way check valve and fill the sampler.

5.3.3.3. Preparation: The steps listed in Section 5.3.1 shall be completed prior to sample collection using low flow methods. The following additional preparatory steps shall be completed:

1. Determine the depth interval at which the HydraSleeve™ sampler will be placed for each well being sampled. Review the HydraSleeve™ manufacturer's SOP for helpful information.
2. Verify the HydraSleeve™ sampler selected will be capable of collecting the sufficient volume of groundwater required for the laboratory for analysis at each well.

5.3.3.4. Groundwater Sampling: Following the preparatory actions described above, the Sample Team shall complete the following steps to collect HydraSleeve™ groundwater samples:

1. Open well and measure depth to water level and total depth of the well using an electronic water level meter. Record these measurements into the project specific log or electronic form.
2. Attach the measured line to the top and a weight to the bottom of the empty sampler and slowly lower the assembly into the well. Avoid any rapid upward movements to prevent water accidently filling the sleeve from the incorrect depth interval.
3. The assembly should be designed to stop on the bottom of the well with the top of the HydraSleeve™ just below the zone intended to be sampled (generally the screened portion of the well).
4. Document in detail the specifics of each well assemble so future sampling can replicate the event.
5. After the HydraSleeve™ sampler has been placed in the well, secure the tether at the wellhead ensuring the HydraSleeve™ sampler in the well is not moved or disturbed.
6. Check and record the depth to water in the well. If needed, allow time for the water level in the well to recover to within approximately 10-percent or less of the depth to water as was measured prior to sampler placement.
7. For sample recovery, the HydraSleeve™ sampler will be activated by gripping the tether at the wellhead, keeping the tether taught, and in one smooth motion, pull the sampler upward at a constant rate of 1 to 2 feet of rise per second through the zone of interest (or well screen). This action must be done as one movement, without stopping, over the length of the sample interval desired.
8. If insufficient sample volume is collected when the HydraSleeve™ is retrieved, a new HydraSleeve™ will be deployed and the procedure will be repeated.
9. To transfer a sample from the HydraSleeve™ with the least amount of aeration and agitation, use the short discharge tube included with the sampler.
   (a) First, squeeze the full sampler just below the top to expel water above the flexible reed-valve.
   (b) Then push the pointed discharge tube through the outer polyethylene sleeve about 3-4 inches below the white reinforcing strips. Discharge the sample into the sample containers.

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(c) Raising and lowering the bottom of the sampler or pinching the sample sleeve just below the discharge tube will control the flow of the sample. The sample sleeve can also be squeezed, forcing fluid up through the discharge tube.

10. Arrange the sample containers in the order of use. VOCs first, if required, SVOCs second, if required, followed by all other samples.
11. Label each sample container with sample ID, date, time, analysis, and other information required on the sample label. Immediately place the filled containers in the coolers(s) on ice.
12. Record sample types, amounts collected, time, and date of collection in the field logbook and on the monitoring well purge and sample log (**Exhibit 1**).
13. Perform post-sampling activities (Section 5.3.5).

5.3.4. Direct Push Groundwater Sampling

5.3.4.1. The direct push groundwater sampling method (also referred to as Hydropunch) is used to acquire groundwater samples from the most permeable zones (sand and gravel layers and lenses) at lower costs than the drilling and installation of groundwater monitoring wells. Chemical analysis of the groundwater samples will provide information about the distribution of contamination and can aid in effectively locating permanent monitoring wells at the site. The techniques are intended to provide the following information:

- Confirmation of potentially contaminated source areas identified during previous studies.
- Groundwater data downgradient of suspected contaminant sources.

5.3.4.2. Preparation: The steps listed in Section 5.3.1 shall be completed prior to sample collection using low flow methods. The following additional preparatory steps shall be completed:

1. The Sample Team Leader will obtain any necessary excavation permits and, if necessary, contact a local underground utility locating service to perform a utility clearance for all borehole locations.
2. If MEC hazards are present, the MEC Escort shall practice anomaly avoidance (see Section 5.2.4).

5.3.4.3. Groundwater Sampling: Following the preparatory actions described above, the Sample Team shall complete the following steps to collect Hydropunch groundwater samples:

1. Hydropunch groundwater samples will be collected using a Geoprobe® or similar direct push drill rig operated by an appropriately licensed driller.
2. The driller will assemble the groundwater sampling device:

   (a) The sampling device will consist of a 52 inch rod with 1.5 inch outside diameter (OD) and alloy steel encasing a stainless steel screen (1–inch OD and 0.004–-inch slot opening).
   (b) An expendable drive point is placed in the lower end of the sampler sheath while a drive head is attached to the top.
   (c) Alternate sampling equipment may be utilized based on direct push equipment in use.

3. The driller will thread the groundwater sampler onto the leading end of the probe rod and drive into the surface with the direct push rig, adding probe rods as needed until the target sample depth is reached
4. The driller will then use extension rods with a screen push adapter to release the expendable point breaking the seal and allowing water to fill through the screen and into the sampler.
5. The tool string and sheath may be retracted the full length of the screen or as little as a few inches if a small sampling interval is required.

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6. Groundwater samples are obtained with a peristaltic pump using 0.25–inch OD polyethylene tubing down the probe rod string into the stainless steel screen and pumping the water to the surface. A bailer may used for retrieving groundwater from depths greater than 25 feet.
7. If the sample water is muddy, purging of the well may be conducted to attempt to get a less turbid sample. Verify in the approved work plan if purge water needs to be containerized and treated as IDW.
8. Collected Groundwater directly into laboratory provided sample containers. Measure and record water quality parameters if required by the approved work plan.
9. Arrange the sample containers in the order of use. VOCs first, if required, SVOCs second, if required, followed by all other samples.
10. Label each sample container with sample ID, date, time, analysis, and other information required on the sample label. Immediately place the filled containers in the coolers(s) on ice.
11. Record sample types, amounts collected, time, and date of collection in the field logbook and on the monitoring well purge and sample log (**Exhibit 1**).
12. Hole abandonment will consist of filling the bore hole with bentonite product or grout. If the bore hole collapses during the removal of the rods the remainder of the open hole will be grouted to ground surface.
13. Perform post-sampling activities (Section 5.3.5).

5.3.5. Post Sampling Activities for GroundwaterSampling

The following steps shall be completed once groundwater sample collection is complete:

1. The Sample Team Leader or designee will confirm the required samples have been collected, including necessary QC samples as specified in the approved work plan.
2. The Sample Team Leader or designee shall record the sample location GPS coordinates.
3. The Sample Team will decontaminate reusable sampling equipment as described in Section 5.4 or as specified in the approved work plan.
4. The Sample Team Leader or designee shall complete the CoC and other required documentation (see Section 5.2.1) and prepare the sample for shipment (see Section 5.2.2). *One trip blank per cooler is required if groundwater is to be analyzed for VOCs*. Trip blanks will be supplied by the laboratory and will be analyzed only for VOCs. Other QC samples will be collected as specified in the approved work plan.

5.4. Sampling Equipment Decontamination

5.4.1 Disposable equipment shall be used wherever possible to limit the potential of cross-contamination. However, if reusable equipment is used (e.g. direct push tooling or cutting shoes), unless otherwise specified in the approved work plan, sampling equipment will be decontaminated using the following process:

1. Decontamination shall be conducted in an uncontaminated area free of dust.
2. Wash equipment with tap/potable water and laboratory-grade detergent (e.g., Alconox™ or Liquinox™). A scrub brush will be used to remove any dirt and/or surface film.
3. Rinse equipment thoroughly with tap water.
4. Rinse equipment thoroughly with ASTM Type II water.
5. Remove excess water and allow equipment to dry.
6. Wrap equipment in aluminum foil, shiny side out.

5.4.2 If required by the Waste Management Plan in the approved work plan, sampling equipment decontamination water shall be containerized for subsequent chemical analysis and for proper disposal of decontamination water. Equipment blanks shall be collected as specified in the approved work plan.

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6. REFERENCES

Reference Title (Author)	Brief summary of relevance to this procedure
ASTM Practice D 6771-02 :Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations. American Society for Testing and Materials, February 2002.	This practice covers the method for purging and sampling wells and devices used for ground-water quality investigations and monitoring programs known as low-flow purging and sampling.

7. EXHIBITS

Exhibit 1: Low-Flow Groundwater Purge and Sample Log

Exhibit 2: HydraSleeve™ Sample Log

Exhibit 3: Direct Push (Hydropunch) Groundwater Sample Log

REVISION HISTORY

Rev.	Date	Summary of Changes	Reason for Revision
00	02/18/15	Initial Release	n/a

EXHIBIT 1

LOW-FLOW GROUNDWATER PURGE AND SAMPLE LOG

Project No.:	Site ID:	
Installation:	Log Book No.	Pages:
Contractor:	Sampler(s)	
Purge Start Date: / / Time:	Purge End Date: / / Time:	
Weather: Wind mph Precipitation:	Air Temperature: °F	
Well Labeled: Y/N [ ] Well Secure: Y/N [ ]	Comments:	
PID SN:	Well Headspace (PID mu) Odor	
Water Level Instrument:	Serial No.:	
SWL beginning (BTOC):	WL After pump install (BTOC):	Max Drawdown (inches):
Well Casing 2" 4" 6" Other:	Borehole diameter:	Sandpack length (L): ft.
Screen Length:	Parameters Measured With:	
Water Column height (h): ft.	Total Purge Vol. Gallons	
Purge Method:	Max Purge Rate: L/min	Sampling Flow Rate: mL/min
Pump Type:	Pump Vol.:	Tubing Material: / Vol./ft: / Total ft.:
Flow-Through Cell Vol.:	Total Pump + Tubing + Cell Vol.:	
Casing radius:_____(in)/12 = _____ **r** (decimal ft)	Borehole radius:_____(in)/12 = _____ r (decimal ft)	
Well Casing Vol. = 3.14 x **r**(_____)^2 x h(_____) x 7.48 (conversion from ft^3 to gal.) = gallons		
Sandpack Vol. = 3.14 x r(_____)^2 x L(_____) x 7.48 – Well Casing Vol.(_____above) x 0.3 = gallons		
Total Well Vol. = Well Casing Vol. (_____above) + Sandpack Vol. (_____above) = gallons		
Depth of pump inlet (BTOC) and rational:		

PURGE CYCLE

Actual Time	Elapsed Time	Volume Purged (gals)	Depth to Water (ft)	Depth of Pump Intake (ft)	Temp (°F)	pH	DO	ORP mV	Conductivity (µmhos/cm)	TDS ppm	Turbidity (NTU)	Comments

SAMPLE

Actual Time	Elapsed Time	Volume Purged (gals)	Depth to Water (ft)	Depth of Pump Intake (ft)	Temp (°F)	pH	DO	ORP mV	Conductivity (µmhos/cm)	TDS ppm	Turbidity (NTU)	VOC Collection Flow Rate

Sample Type:	Sample No.	
Sample Equipment	Sample Filtered: Yes [ ] No [ ]	Filter Type/Size:
Equipment Rinsate Sample No.:	Sample Equipment Decon: Date: by:	
Comments:		
Discharge Water Disposition:	Drum Number:	
Prepared by: Date: / /	Reviewed by: Date: / /	

PURGE CYCLE (CONTINUED)

Actual Time	Elapsed Time	Volume Purged (gals)	Depth to Water (ft)	Depth of Pump Intake (ft)	Temp (°F)	pH	DO	ORP mV	Conduct ivity (μmhos/ cm)	TDS ppm	Turbidity (NTU)	Comments

EXHIBIT 2

HYDRASLEEVE SAMPLE LOG

HYDRASLEEVE DEPLOYMENT

Project No.:	Well LOCID:	
Installation:	Log Book No.	Pages:
Contractor:	Sampler(s):	
HS Deployment Date: / / Time:	Weather: Wind Dir: , at ~___mph; Air Temp: °F	
Well Labeled: Y/N [ ] Well Secure: Y/N [ ]	Comments:	
PID SN:	Well Headspace (PID mu): Odor:	
Water Level Instrument:	Serial No.:	

SWL (ft BTOC):	Measured Well Depth (ft BTOC):	Reported Well Depth (ft BTOC):
Sediment Thickness (ft):	Number of Hydrasleeves deployed in well:	Tether Line Material: ☐Polypropylene Rope ☐Stainless steel
Type of Tether Weight:	Total Weight used (oz.):	
Sleeve bag length (in):	HS bag volume (ml):	Depth to top of sleeve (ft BTOC):
Bottom Weighted: Y/N [ ]	Top Weighted: Y/N [ ]	

HYDRASLEEVE RETRIEVAL AND SAMPLE

Well LOCID:	Hydrasleeve Retrieval Date: Retrieval Time:	
Log Book No.	Pages:	
Was ALL Deployed Equipment Retrieved (Line, Bags, Weights): Y/N [ ] if NO, Explain:		
Comments on Well and Hydrasleeve Tether Assembly Condition:		
Weather: Wind Dir: , at ~_____mph; Precipitation:	Air Temperature: °F	
Sample No. (FIELDSAMPID):	Sample Date: / /	Sample Time:
Sampler (s):	Sample Beg. Depth (ft BTOC):	Sample Ending Depth (ft BTOC):
Sample Collection Method: ☐Discharge Tube ☐Other (explain):		
Approximate Volume of Excess Sample Water After Sampling (ml):		
Excess Sample Water Placed in Container: Y/N [ ]	Container Number:	
SWL Following Sampling (ft BTOC):	Sample Equipment Decon Date: by:	
Decon Water Placed in Drum: Y/N [ ]	Drum Number:	
Prepared by: Date: / /	Reviewed by: Date: / /	

EXHIBIT 3

DIRECT PUSH (Hydropunch) GROUNDWATER SAMPLE LOG

Sample ID:	Page 1 of ____	
Project:	SWMU: Nearest IDF:	
Installation:	Log Book No.:	Log Book Pages:
Contractor:	Sampler Name:	
Direct Push Subcontractor:	Driller Name:	
Drill Start Date: / / Drill Start Time:	Drill End Date: / / Time:	
Sample Date: / / Sample Time:	Water Parameters Measured : YES NO	
Sample Depth (Ft BGS):	Approx Depth to Water (Ft BGS):	
Sample Method: Peristaltic Pump Bailing Other_____		
Purge Water Disposition IDW Drum No.:		

WATER PARAMETERS

Time	Volume Removed (gal.)	Turbidity (NTUs)	Clarity/Color	DO (mg/l)	ORP (mV)	Temp. (°C)	pH	CONDUCTIVITY (mS/cm)	Remarks:

Water Quality Meter	Turbidity Meter
Type/Model:	Type/Model:
Serial No. :	Serial No. :
Calibration Date: / /	Calibration Date: / /

NOTES/REMARKS

Appendix B

Field Sampling Forms

GROUNDWATER ELEVATION REPORT

PARSONS	CLIENT:	DATE:
PROJECT:		PROJECT NO:
LOCATION:		INSPECTOR:

MONITORING EQUIPMENT:

INSTRUMENT	DECTECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR:

INSTRUMENT	CORRECTION FACTOR

COMMENTS:

WELL	TIME	DEPTH TO WATER	DEPTH TO PRODUCT	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC. GRAV.	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of: riser, concrete, protective casing, etc.)

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #:

PROJECT:

LOCATION:

DATE:

INSPECTORS:

PUMP #:

SAMPLE ID #:

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)

SAMPLING ORDER			PRESERVATIVES		BOTTLES		SAMPLE NUMBER	TIME	CHECKED BY/
					COUNT/ VOLUME	TYPE			DATE

COMMENTS: (QA/QC?)

IDW INFORMATION:

Ash Landfill Conditions Survey

Location	NOTES (include date)
NCFL	
Ash LF	
Biowall A1/A2	
Biowall B1	
Biowall B2	
Biowall C1	
Biowall C2	
ZVI wall	
Well conditions	

OB Grounds

Date of Inspection:
Weather Conditions:

Observations should include assessment of integrity of 9-inch soil cap placed over residual lead contaminated soil in 25 125'x125' grids.

Assessment should be made with respect to caps ability to ensure that indegenous terrestrial wildlife are not exposed via direct dermal contact or incidental ingestion.

Note signs of erosion or animal burrowing to ensure underlying soils are not exposed to the environment.

	Grid No.	Observations/Location of Disturbed Soils
1	A5	
2	C5	
3	B3	
4	B2	
5	C3	
6	C2	
7	C1	
8	C7	
9	D7	
10	E9	
11	H9	
12	I6	
13	I7	
14	I8	
15	J5	
16	J6	
17	J8	

	Grid No.	Observations/Location of Disturbed Soils
18	L8	
19	L9	
20	L10	
21	M10	
22	N10	
23	P10	
24	Q7	
25	Q8	
26	R8	
27	S8	

Appendix C

Analytical SOPs

Analytical SOPs are provided on the electronic (CD) version of this report.

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-202
Revision History
Cover Page
Page 1

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Prepared By: GC/MS Group Date: 2/97

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1.23.01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8260B	Format changes, added pollution prevention, changes to calibration section, new limits, added instrument. Other minor changes throughout.	DN	1.23.01	1.23.01
04 8260B	Revised sections 7.5.3.1, 7.5.5, 7.7.1, 7.8.2 & Table 2 to comply with South Carolina. Added NH oxygenates to calibration.	DN	5.23.01	5.23.01
05 8260B	Updated VOA calibration standard mixes. Added statistical limits for LCS/MS/MSD recoveries and the updated corrective actions	DN	5.21.02	5.21.02
06 8260B	Reorganization of sections 4, 5, 6 and 7, and Tables and Figures. Added definitions and information for the new data processing system.	MRC	05.03.04	05.03.04
07 8260B	Minor changes rewording of sec 1.7.6.3 preservation of calcareous soils	LAD	020305	020305

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8260B	Added references, setup and operation for the Encon / Centurion autosampler / Purge and Trap. Added ref. to instrument "T" and removed instrument "Q". Edited Std. conc. to reflect new instrumentation. Minor changes throughout to reflect current practice and correct typos.	LAD	04/06	04/06
09 8260B	Sect. 11 - added list of waste streams generated and location of satellites. Clarified RT window studies. Added reference to MI Sop. Removed Grand Mean Calibration model. Added wording for project specific acceptance criteria. Added LCS marginal outlier criteria. Added wording clarifying Calibration verification Std. Criteria and corrective action. Reworded Correlation	LAD	LAD 7-25-07 ~~03/07~~ 07/07	~~03/07~~ 07/07
10	Coefficient criteria Updated Sections 7.4.5, 7.4.6, 7.4.7, 7.5.2, 8.1, 10.0 and Table 1 with DoD QSM Version 4.1 criteria	LAD	08/09	08/09
11	Added Table 2 with DoD QSM V. 4.1 QC Requirements. Added if the MSD Batch requirement can not be fulfilled, a LCSD must be analyzed. Removed "2" instrument and added the "C" and "D" instruments.	LAD	04/10	04/10
LAD 05/11/11 12	Removed Tekmar 2000 and 2016 throughout. Sect. 7.3.1 - Removed ~~5970~~ 5970 GC/MS instrument type. Sect. 7.4.7 - Added RRT information. Sect. 8.1 - Added S.C. marginal exceedence criteria. Sect. 9 - Added MDL, LOD and LOQ Criteria. Updated figures.	LAD	05/11	05/11
13	Sect. 5 - Changed Cal Mix and ICV Std. Exp. from 7 to 14 days. Sect. 6 - Add Sample preservation info. Sect. 7.4.4.1 - Added S.C. exemption from 2nd order cal. Sect. 7.5.1 - Added Extras mix to LCS. Sect. 7.6.12 - clarified Nothing why samples need to be reanalyzed. Sect. 8.1 - Added 10% rule for LCS, ICV and MS/D. Sect. 9 - Added LOD/LOQ definitions. Table 1 - Reworded CA for ICV.	LAD	03/12	03/12
14	Sect. 1 and 7 - Removed Quickform references and added reporting from KIMS. Sect. 7 - Removed Soil 200 ug/kg level and added 80% level. Sect. 8 - Added additional marginal exceedance information. Throughout - Fixed typos and made minor edits.	LAD	04/13	04/13
15	Sect. 4 - Removed 5890, 5972 and Tekmar references. (5970 too). Sect. 10 - Updated and added references. Table 3 - Added DoD QSM ver. 5.0 QC requirements. Renumbered Tables 3, 4, 5.	LAD	04/14	04/14

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
16	Sect. 1 – Added sand to m. bik def. Sect. 5 – Separated cal std into VOA gases and non-gases, clarified VOA gases exp. date, combined Surr & IS std, added sand. Sect. 6 – Added Acrolein and Acrylonitrile preservation. Sect. 7 – Added poor purgers % RSD calibration info, added CCV criteria, added use of sand in soil Blank and LCS's, updated cal spike prep. Sect 8 – Added additional LCS criteria. Updated Figures 1 – 3.	LAD	07/16	07/16

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-202-16**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-202-16**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: _____ Date: _____

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze aqueous and solid matrix samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision.

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

1.1 Definitions

VOC: Volatile Organic Compounds

VOA: Volatile Organic Analysis

ANALYTICAL BATCH: 20 or fewer samples that are analyzed together with the same method sequence and the same lots of reagents and with the handling practices common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. This is prepared as an LCS and analyzed after the calibration before any sample analysis.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions containing target analytes are added to a sample matrix prior to sample extraction, in the case of soils, and/or analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of volatile organics by the current revision of EPA Method 8260. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Demonstration of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of volatile organics by Method 8260 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded

in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin’s disposal policies.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

There are three general types of waste generated while performing the 8260 method. The "K" waste is a combination of water, sample aliquot (post analysis), as well as internal and surrogate standards. "K" waste is generated when preparing QC, during sample analysis, and procedural cleanup. There are "K" satellites attached to each GC/MS instrument as well as an additional satellite located adjacent to the VOA sample preparation bench. "O" waste consists of methanol (as well as trace amounts of volatile analytes) and is generated when standard preparation syringes are rinsed three times with methanol. The "O" waste stream satellite is located inside the fume hood. Organic soil waste stream "I" consists of any solid left over from sample preparation and/or analysis and is located inside the fume hood. All satellites listed above are stored in a secondary container and are located in the Volatile Organics Laboratory room 111.

2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous and soil samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. Soil samples with higher contaminant levels are extracted with methanol prior to the helium purge. These volatile organics are then retained on a cooled trap (commercially available trap suitable for the methodology) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration. Typically 2 or 3 rinsing blanks are analyzed at the end of a sequence. Samples are not analyzed on the instrument until a blank with no detects above PQL can be obtained. If the lines are determined to be contaminated, then the entire concentrator must be backflushed with warm methanol and water.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph (GC): Hewlett Packard 6890.

4.2 Mass Spectrometer (MS): HP5973

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Column: RTX-VMS, 40 meter, 0.18 mm ID or equivalent.

4.5 Purge and Trap: Archon or Centurion auto samplers, and Encon concentrators.

4.6 Purge tubes: 5 mL fritted and 25 mL fritted purge vessels and 40 mL VOA vials for soil analysis.

4.7 Hamilton Gastight syringes: 2.00 uL to 25.00 mL.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS AND STANDARDS

5.1 Purge and trap grade methanol

5.2 Organic-free Laboratory reagent grade water: Siemens, Poland Spring, or equivalent. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”. After ampulated standards are cranked open, the standard is transferred to a screw top vial and stored in a freezer.

5.3.1 The expiration date for all standards except volatile gases is six months from date of opening the ampule.

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 Calibration Mix (without gases) – Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 100 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 14 days and stored in the VOA standards freezer between uses.

1,2-Dibromo-3-chloropropane	2,2-Dichloropropane	Dibromomethane
1,1,1,2-Tetrachloroethane	2-Butanone	Ethylbenzene
1,1,1-Trichloroethane	2-Chloroethylvinyl ether	Hexachlorobutadiene
1,1,2,2-Tetrachloroethane	2-Chlorotoluene	Idomethane
1,1,2-Trichloroethane	2-Hexanone	Isopropylbenzene
1,1-Dichloroethane	4-Chlorotoluene	Methyl tert-butyl ether
1,1-Dichloroethene	4-Methyl-2-pentanone	Methylene chloride
1,1-Dichloropropene	Acetone	Naphthalene
1,2,3-Trichlorobenzene	Benzene	n-Butylbenzene
1,2,3-Trichloropropane	Bromobenzene	n-Propylbenzene
1,2,4-Trichlorobenzene	Bromochloromethane	p-Isopropyltoluene
1,2,4-Trimethylbenzene	Bromodichloromethane	sec-Butylbenzene
1,2-Dibromoethane	Bromoform	Styrene
1,2-Dichlorobenzene	Carbon disulfide	tert-Butylbenzene
1,2-Dichloroethane	Carbon Tetrachloride	Tetrachloroethene
1,2-Dichloropropane	Chlorobenzene	Tetrahydrofuran
1,3,5-Trimethylbenzene	Chloroform	Toluene
1,3-Dichlorobenzene	cis-1,2-Dichloroethene	trans-1,2-Dichloroethene
1,3-Dichloropropane	cis-1,3-Dichloropropene	trans-1,3-Dichloropropene
1,4-Dichlorobenzene	Cyclohexane	Trichloroethene
1-Chlorohexane	Dibromochloromethane	Vinyl Acetate

5.3.2.2 Gases Calibration Mix - Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 100 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

Bromomethane
Chloromethane
Dichlorodifluoromethane
Trichlorofluoromethane
Vinyl Chloride
Chloroethane

5.3.2.3 Extras mix – Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 100 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

Acetonitrile	Isobutyl alcohol
Acrolein	Methacrylonitrile
Acrylonitrile	Methylcyclohexane
Allyl chloride	Methyl acetate
Chloroprene	Methyl methacrylate
Diethyl ether	Methyl tert-butyl ether
cis-1,4-Dichloro-2-butene	Pentachloroethane
trans-1,4-Dichloro-2-butene	Propionitrile
1,4-Dioxane	Tertiary-amyl methyl ether
di-Isopropyl Ether	Tertiary-butyl alcohol
Ethyl methacrylate	1,3,5-Trichlorobenzene
Ethyl tertiary-butyl ether	1,2,3-Trimethylbenzene
Freon-113	

5.3.2.4 Independent Calibration Verification Standard, Laboratory Control Spike and MS/MSD Mixture - Prepare a standard as above containing the compounds listed in Table 3. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 14 days and stored in the VOA standards freezer between uses.

5.3.2.5 Surrogate/Internal Standard Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

Internal Standards	Surrogate Standards
Pentafluorobenzene	4-Bromofluorobenzene
1,4-Difluorobenzene	1,2-Dichloroethane-D_4
Chlorobenzene-D_5	Toluene-D_8
1,4-Dichlorobenzene-D_4	Dibromofluoromethane

5.3.2.6 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 25 ug/mL. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

NOTE: The concentrations of standards may vary depending on the type of autosampler being used.

5.4 Organic Free Sand – Ottawa Sand or equivalent baked at 110 ºC overnight

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous samples

All aqueous samples are collected in 40 mL VOA bottles with no headspace, preserved with 1:1 HCl to a pH of <2 and stored at <6°C until analysis. Aqueous samples must be analyzed within 14 days from sample collection if preserved and within 7 days from sample collection if unpreserved.

Samples requiring Acrolein and Acrylonitrile analysis, require preservation of pH of 4-5 and cool to 0-6ºC.

6.2 Soil Samples

Soil samples arriving at the laboratory in Terra-core or Encores Soil samplers must be extruded into water or sodium bisulfate within 48 hours of sampling. Soils samples extruded into water must be frozen at -15°C ± 5°C until analysis. Soil sample extruded into sodium bisulfate must be stored at <6 °C until analysis.

Medium level soil (methanol preserved) samples are sampled into pre-weighed vials containing 5 mLs methanol. Methanol preserved soil samples must be stored at <6°C from the time of receipt at the lab until analysis.

Bulk soil samples are stored at <6 °C until analysis.

All soil/sediments must be analyzed within 14 days from sample collection.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS - Conventions for all instruments are as follows:

- Sub-Directory for data acquisition: C:\HPCHEM\1\DATA
- Tune file: BFB.U
- Method files:
  For BFB Tune: BFB288AQ.M (waters) or BFB288SL.M (soils)
  For all samples and standards: I826AXX.M
  where: I = instrument ID (Each instrument is given a unique identifier).
  A = matrix (A for water, S for soil and SB for sodium bisulfate soils)

XX = the calibration number in chronological order

- Data files:

  For BFB: IB_ _ _.D

  where: I is the instrument ID
  _ _ _ is a number in chronological order from 000 to 999.

  For all other data files: I_ _ _ _.D
  Where: I is the instrument ID
  _ _ _ _ is a number in chronological order from 0000 to 9999.

  This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50 ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:

Mass	Criteria
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

7.3.1 The following are the GC/MS operating conditions for injection of BFB.

Column:		RTX-624, 40 meter, 0.18 mm I.D or RTX-VMS, 40 meter, 0.18 mm ID.
Temperatures:	Injection port:	200°
	Transfer line:	150°
	Detector:	240°
Isothermal temperature:		150°
Run time:		8 minutes
Scan start time:		3 minutes
Scan parameters:		not to exceed 2 sec per scan
Mass range:		35-300
Number of A/D samples:		8
GC peak threshold:		1000 counts
Threshold:		10 counts

The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 12-hour clock. All

calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB run has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

7.4 INSTRUMENT CONFIGURATION / CALIBRATION

Purge and Trap conditions:

Parameter	Aqueous	Soil
Standby:	35°	35°
Prepurge:	0 min	0 min
Preheat Temp:	Ambient	40°
Sample Temp:	Ambient	40°
Purge:	11 min	11 min
Purge Flow Rate	~24-40 mL/min	~24-40 mL/min
Dry purge:	2-4 min	2-4 min
Desorb preheat:	245°	245°
Desorb Temp:	250°	250°
Desorb Flow Rate:	~15 mL/min	~15 mL/min
Desorb time:	2-5 min	2-5 min
Dry purge:	2-4 min	2-4 min
Bake Time:	10 min	10 min
Bake Temp:	260°	260°
Auto drain:	On	On
Bake gas by pass:	Off	Off
Valve Temp:	120°	120°
Line Temp:	120°	120°
Runs per sample:	1	1

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

Please refer to the Encon, Archon and Centurion Opperating manuals for more specifics on programming features.

7.4.3 Initial Calibration for Method 8260

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

To determine the linearity of response, the GC/MS must be initially calibrated at six different levels.

For aqueous calibration, target analytes and surrogate are prepared at the following concentrations; 1.0, 5.0, 20, 50, 100 and 200 ug/L. The curve is analyzed at ambient temperature.

For a soil calibration target analytes and surrogates are prepped at the following concentrations: 5.0, 10, 20, 50, 80 and 100 ug/L. The calibration standards are stirred and heated to 40ºC.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water in order to generate a 6-point initial calibration curve:

Notes	STD. ID	CAL. Mix 100 ug/mL	Extras Mix 100 ug/mL
AQ curve only	VSTD001	1 uL	1 uL
	VSTD005	5 uL	5 uL
SL curve only	VSTD010	10 uL	10 uL
	VSTD020	20 uL	20 uL
CCV	VSTD050	50 uL	50 uL
SL curve only	VSTD080	80 uL	80 uL
	VSTD100	100 uL	100 uL
AQ Curve only	VSTD200	200 uL	200 uL

The Surrogate & Internal Standard is spiked by the autosampler. The Archon Surrogate/IS Mix is at 250 ug/ml and the instrument spikes 1 ul. The Centurion Surrogate/IS Mix is prepared at 50 ug/ml and the instrument spikes 5 ul.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 3 and 5.

7.4.4 Initial Calibration Criteria

The percent (%) RSD for six calibration check compounds (CCC) must be less than or equal to 30%. CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride.

A system performance check must be performed as part of initial calibration. The five system performance check compounds (SPCC) and the minimum acceptable average relative response factors (RRF) for these compounds are as follows (taken from 8260B):

SPCC	RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

The SPCCs are used to check both the standard and instrument stability.

7.4.4.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less using the average response factor, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15% using the average response factor, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target calculates the correlation coefficient and then squares it (r^2). This is what is reported on all Target forms. The value for r^2 must be greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

Note 1: For poor purging compounds like acetone, the %RSD value may exceed the method acceptance limit of 15% but meet the acceptance criteria for the linear and quadratic calibration models. The average calibration model should still be used because this calibration model is more accurate at concentrations near the LOQ than either the linear or quadratic calibration models.

This is common for acetone but also may apply to other poor purging ketones.

In any instance the % RSD must be below 30%.

Note 2: Non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration for compliance work originating in their state. In these cases, a linear calibration model must be used.

7.4.5 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, internal standards and surrogates at a concentration of 50 ug/L and is obtained from a source independent of the initial calibration source. Please refer to section 8.1 and Table 1 for acceptance criteria and corrective action for this standard.

For projects or clients requiring DoD QSM, current revision, all project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria are met.

7.4.6 Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing all the target compounds, internal standards and surrogates at a concentration of 50 ppb must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB. The relative response factor from the 50 ppb continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extracted ion current profile) area for any of the internal standards in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for any internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.

For Method 8260, if the percent difference for each CCC is less than or equal to 20%, and all of the SPCCs have a relative response factor greater than or equal to those listed in Section 7.4.4, the continuing calibration is considered valid.

For projects or clients requiring DoD QSM, current version, all project analytes must have $\pm$ 20%D.

For all other projects, all project analytes should have $\pm$ 30%D ($\pm$ 40%D for poor performers).

Continuing calibration check criteria must be met before sample analysis can proceed.

7.4.7 Retention Time Windows

Retention time windows for each analyte and surrogate are set at the midpoint standard of the calibration curve, following every ICAL. On days when an ICAL is not performed, the initial CCV may be used. For each sample, the RRT shall be compared with the mid-point of the ICAL or the most recently updated RRT. If the RRT has changed by more than ± 0.006 RRT units indicates a significant change in system performance and the laboratory must take appropriate corrective action.

7.5 QUALITY CONTROL SAMPLE ANALYSIS

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

7.5.1 Laboratory Control Sample (LCS)

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the water and medium-level soil LCS, 25 uL of the LCS and Extras standard mix at 200 ug/mL are spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L. The Archon autosampler adds 1 uL of internal and 1 uL of surrogate standard to a 5 mL aliquot of this preparation for analysis. The Centurion autosampler adds 5 uL of both surrogates and internal standards to a 5 mL aliquot. To prepare the low-level soil LCS, a stir bar is added to 5 mL of the above solution plus 5 g baked Ottawa sand, in a VOA vial. The Archon unit adds an additional 10 mL of water to which the internal and surrogate standards have been added; this preparation is then heated, stirred and purged.

NOTE: In the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory Control Spike Duplicate must be analyzed.

7.5.2 Method Blank Analysis

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.

The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The low-level soil method blank is a volume of analyte free laboratory reagent grade water plus 5 g baked Ottawa sand, spiked with internal and surrogate standards. This method blank is analyzed using the low soil specification.

The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM, current version, no analytes may be detected >1/2 the PQL and > than the 1/10th the measured amount in any sample or 1/10th the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected > than the PQL.

7.5.3 Surrogate Recovery Limits

Laboratory established limits are derived for each of the surrogates. Please refer to the current revision of Katahdin Analytical Services SOP # QA-808 for further information on statistical limits. All samples including blanks, laboratory control samples, matrix spikes and client samples, must meet the statistical limits for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

7.5.4 Internal Standard Area Recoveries / Retention Times.

The internal standard responses and retention times in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extracted ion current profile) area for any of the internal standard changes by a factor of two (-50% to +100%), from the last daily calibration standard, the GC/MS must by inspected, and corrective action taken. If the retention time for any internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must be inspected and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

For projects or clients requiring DoD QSM, current version, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

7.5.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner similar to the LCS, except that 40 mL aliquots (aqueous) or 5 g aliquots (soil), of environmental samples are used in

place of the analyte-free laboratory reagent grade water. Note that trip blanks and field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration of 50 ppb. Acceptance criteria for the MS/MSD are outlined in Section 8.0.

NOTE: In the event that sufficient volume of sample is not supplied to the laboratory so that an MS/MSD set cannot be analyzed within a batch of 20 samples, a laboratory control spike duplicate must be analyzed.

7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. He/she may be able to provide more information about the sample. Sample history is used to determine what order in which to run the samples and at what dilution. Refer to Katahdin Analytical Services SOPCA-106, “Basic Laboratory Technique”, current revision for information on subsampling.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

7.6.1 SAMPLE ANALYSIS FOR 8260B WATER

7.6.1.1 Archon Autosamplers

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume and or dilution for the sample. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.

7.6.1.2 Centurion Autosamplers

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the Centurion to run as many samples that will fit into a 12 hour clock. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted or duplicated, it must be noted in the comments section of the injection logbook. Additional information may be needed to assure that any questions that arise during the review process can be answered.

To minimize carryover from samples that contain a target compound at a level exceeding the upper limit of the calibration curve, the following must be done: monitor samples analyzed after the contaminated sample as well as the next run of the contaminated sample in the same purge inlet for the target(s) in question; both must have levels <PQL.

7.6.2 ANALYSIS OF LOW-LEVEL SOIL SAMPLES

Method 5035 Closed System Purge & Trap procedure for low level soils (5 ug/Kg -200 ug/Kg)

Selecting the appropriate technique may depend on cleanup goals, confidence levels, and anticipated levels of contamination. Field sampling activities typically result in Encore or Encore-like devices being submitted to the lab. These devices must be extruded within 48 hours. It is the laboratory's standard policy to extrude soil samples into 5 mL of Laboratory reagent free laboratory reagent grade water that contains a magnetic stir bar. The sample is subsequently frozen until analysis within 14 days. Note that the sample must be extruded and frozen within 48 hours of sampling, until analysis can begin. This approach is preferred over extrusion into sodium bisulfate because it is believed that the sodium bisulfate reacts with calcium carbonate in highly calcareous soils causing effervescence and driving the volatile analytes out of solution. There is also anecdotal information to suggest that acetone may be generated when bisulfate preservation occurs. The Katahdin sample ID, extrusion date, and time are recorded in the GC/MS extrusion logbook. Please refer to the Katahdin method 5035 SOP, CA-214 for more detail.

In lieu of the use of Encore samplers, the lab may pre-weigh 40 mL VOA vials containing 5 mL of laboratory reagent grade water or a 20% sodium bisulfate solution and a magnetic stir bar and ship these to the field. The vial is assigned a vial specific number prior to shipment to the field. The vial and

weight will be recorded with its vial specific number in the methanol soil logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. The samples must be frozen within 48 hours of sampling, until analysis can begin.

The subsequent analysis is performed on a specially developed autosampler that heats, stirs, and purges the sample simultaneously without exposing the contents of the vial to the atmosphere. This procedure will help to minimize the loss of VOC's due to transport, handling, and analysis and may help minimize ambient lab contribution. The expected detection limits are consistent with the traditional low soil technique from method 5030. The Archon is programmed to heat each vial to 40°C during the purge time. Initiate purging for 11.0 minutes; the sample must be heated to 40°C ± 1°C before purging can begin. If you have questions concerning setting up the autosampler or initiating a GC/MS batch run, consult the Organic Department Manager, or senior chemist within the group.

If the client does not require method 5035, method 5030 for analysis of low-level soils may be followed. In this case, the Archon units may be used for the preparative step.

7.6.2 ANALYSIS OF MEDIUM-LEVEL SOIL SAMPLES

Method 5030 Procedure for higher concentration soils (> 200 ug/Kg)

Higher concentration soils may be sampled as either a bulk sample or field preserved with a water miscible solvent such as methanol. If sampled in an Encore unit, the soil is extruded into methanol upon receipt at the lab.

Bulk Sample- A sample is placed in a glass jar or vial and returned to the lab for extraction and analysis. In this approach the lab takes an aliquot of soil and extracts with purge & trap grade methanol, a portion of the methanol is then analyzed for volatile analytes.

Calibrate the balance properly (See SOP CA-102) and note it in the appropriate logbook. Place 5.0 grams of thoroughly mixed, undecanted soil sample in a 40.0 mL vial. Add 5.0 mL reagent grade methanol. Shake for 2 minutes. Let stand for 3 minutes. Record extraction in soil prep logbook.

Methanol Field Preservation - A 5 gram sample is added to a VOA vial that has been previously charged with purge and trap grade methanol (the volume of methanol is dependent upon client request). The vial with methanol has been previously weighed in the lab and assigned a vial specific number prior to shipment to the field. The vial and methanol weight

will be recorded with its vial specific number in the VOA vial prep logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. A portion of the methanol is then analyzed for volatile analytes.

For analysis on Archon or Centurion autosamplers, add 400 uL of the extract into 20 mL of organic-free laboratory reagent grade water (e.g., Poland Spring or equivalent). IS and SS is added by the Archon and/or Centurion autosampler for analysis. This will give an estimated calibration range between 500-10000 ug/Kg.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is performed by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed.

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed.

7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or ISTD area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalyses.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. An "M" qualifier will automatically be printed on the quantitation report summary.
This manual integration package must then be submitted to the Organic Department Manager or his/her designee, who will review each manual integration.

For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.
If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should

immediately be forwarded to the Organic Department Manager, or his/her designee.

7.7.1.3 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer to SOP CA-207 "GC/MS Library Search and Quantitation".

7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into Kims. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information

will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Independent Calibration Verification, LCS and MS/MSD Criteria

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

The LCS recoveries for all analytes are evaluated. For non-DOD clients, the exceedances from the laboratory established limits or nominal limits must be less than ten percent of the client compound list. For DOD clients, all of the compounds of interest must fall within either Katahdin's statistically derived limits or the DOD QSM, current version, limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They also can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required.

Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

Note: South Carolina does not allow for marginal exceedences for compliance work originating in their state. Additionally, the laboratory statistically derived LCS limits should fall within 70-130%.

The MS/MSD recoveries for all analytes are evaluated. If the LCS results are acceptable but the MS/MSD is not, narrate. If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

For projects or clients requiring DoD QSM, current version, all project analytes in the ICV must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met. Laboratory established recovery limits for LCS and MS/MSDs must be within 3 standard deviations of the mean LCS recovery. MS/MSD pairs must be run once per analytical/preparatory batch. RPDs must be less than or equal to 30% between MS and MSDs.

For analytes with no available DoD acceptance criteria, laboratory established limits shall be used.

8.2 Surrogate Recovery Criteria

Statistical limits are compiled annually for surrogate recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

8.3 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based

on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

Limits of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the anaysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.
Refer to the current revision of Method 8260 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8260B.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.2, 10/25/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration for all a nalytes	Initial calibration prior to sample analysis	SPCCs average RF □0.30, except chloromethane, 1,1-DCA and bromoform ≥0.10; RSD for RFs □ 30% for CCCs. Refer to section 7.4.3 also.	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances.	If the surrogate recoveries in the ICV are low but the target analytes are acceptable, narrate. If the ICV recovery is high but the sample results are <PQL, narrate. If the ICV is out but the batch LCS is in criteria, narrate.
Calibration verification	Once per each 12 hours, prior to sample analysis in absence of initial cal	SPCCs minimum RF ≥ 0.30, except chloromethane, 1,1-DCA and bromoform ≥ 0.10; RF for CCC analytes □ 20% (%D) of average initial multipoint RF	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	During data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Method Blank	One per batch of 20 or fewer samples.	No analytes of interest detected > PQL with the exception of Methylene Chloride	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per batch of 20 or fewer samples.	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances. .	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Statistically derived limits.	Reprep and reanalyze for confirmation of matrix interference when appropriate.
MS/MSD	One MS/MSD per every 20 samples.	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits. .	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL Studies, LOD and LOQ Verifications	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	Once per year for each analyst; 4 reps	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

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TABLE 2

DOD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30% .	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

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TABLE 2

DOD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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TABLE 3

DOD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation = 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Flagging is not appropriate.	The DDT breakdown and Benzidine/Pentachlorophenol tailing factors are considered overall system checks to evaluate injector port inertness and column performance and are required regardless of the reported analyte list.
Initial calibration (ICAL) for all analytes (including surrogates) At instrument set-up, prior to sample analysis	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: Option 1: RSD for each analyte = 15%; Option 2: linear least squares regression for each analyte: r2 = 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 = 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Required for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.

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TABLE 3

DOD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification. Results may not be reported without a valid CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 3

DOD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch. A laboratory must use the QSM Appendix C Limits for batch control if project	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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TABLE 4

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-202-16	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(1) Use laboratory reagent grade water for low level soil calibration, method blanks, and laboratory control samples to minimize clogging of archon soil needles with sand. (2) Internal Standards- pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4	(1) Use an aliquot of a clean (control) matrix similar to the sample matrix. (2) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC - MDL	None	

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TABLE 5

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Acetone	43	58
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39
Benzene	78	-
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
Sec-Butylbenzene	105	134
Tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexane	56	84, 60
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
Diethyl Ether	74	45, 59
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
Cis-1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Cis-1,3-Dichloropropene	75	77, 39
Trans-1,3-Dichloropropene	75	77, 39
Cis-1,4-Dichloro-2-butene	75	53, 77
Trans-1,4-Dichloro-2-butene	53	88, 75
1,4-Dioxane	88	58, 43
Di-Isopropyl ether	45	43, 87

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TABLE 5

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Ethylbezene	91	106
Ethyl methacrylate	69	41, 99
Ethyl tertiary-butyl ether	59	87, 57
Freon-113	151	101
Hexachlorobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Idomethane	142	127, 141
Isobutyl alcohol	43	41, 42
Isopropylbezene	105	120
p-Isopropyltoluene	119	134, 91
Methacrylonitrile	41	67, 39
Methylcyclohexane	83	55, 98
Methylene chloride	84	86, 49
Methyl acetate	43	74
Methyl methacrylate	69	41, 100
4-Methyl-2-pentanone	43	58, 85, 100
Methyl tert-butyl ether	73	57, 41
Naphthalene	128	-
Pentachloroethane	167	130, 132
Propionitrile	54	52, 55
n-Propylbenzene	91	120
Styrene	104	78
Tertiary-amyl methyl ether	73	55, 87, 71
Tertiary-butyl alcohol	59	41, 43
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Tetrahydrofuran	42	72, 71
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,3,5-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,3-Trimethylbenzene	105	120
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
Xylenes (Total)	106	91
1-Chlorohexane	91	55,43

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TABLE 6

ANALYTE QUANTITATION AND INTERNAL STANDARDS

Pentafluorobenze	**1,4-Difluorobenzene**	**Chlorobenzene - d5**	**1,4-Dichlorobenzene - d4**
Dichlorodifluoromethane	1,2-Dichloroethane	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Chloromethane	1,1-Dichloropropene	Tetrachloroethene	1,2,3-Trichloropropane
Bromomethane	Carbon tetrachloride	Dibromochloromethane	Isopropylbenzene
Vinyl chloride	Benzene	Chlorobenzene	Bromobenzene
Chloroethane	1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	2-Chlorotoluene
Trichlorofluoromethane	Trichloroethene	Ethylbenzene	4-Chlorotoluene
Methylene Chloride	Dibromomethane	Xylenes (total)	1,3,5-Trimethylbenzene
Acetone	Bromodichloromethane	Bromoform	Tert-Butylbenzene
1,1-Dichloroethene	cis -1,3-Dichloropropene	Styrene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	4-Methyl-2-pentanone	2-Hexanone	Sec-Butylbenzene
cis-1,2-Dichloroethene	Toluene-d8 (surr.)	Bromoform	1,3-Dichlorobenzene
trans-1,2-Dichloroethene	Toluene		P-Isopropyltoluene
Chloroform	trans-1,3-Dichloropropene		1,4-Dichlorobenzene
2,2-Dichloropropane	1,1,2-Trichloroethane		1,2-Dichlorobenzene
2-Butanone	1,2-Dibromoethane		N-Propylbenzene
Methyl-tert-butyl ether (MTBE)	Vinyl Acetate		1,2-Dibromo-3-chloropropane
Tetrahydrofuran	Methyl Methacrylate		1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl Methacrylate		Naphthalene
1,1,1-Trichloroethane	1,4-Dioxane		Hexachlorobutadiene
Tertiary-butyl alcohol (TBA)	2-Chloroethylvinyl ether		1,2,3-Trichlorobenzene
Di-isopropyl ether (DIPE)	Bromofluorobenzene (surr.)		cis-1,4-Dichloro-2-butene
Ethyl-tert-butylether (ETBE)			trans-1,4-Dichloro-2-butene
Tertiary-amyl methyl ether			Pentachloroethane
Diethyl ether			n-Butylbenzene
Carbon disulfide			1,3,5-Trichlorobenzene
Freon-113			1,2,3-Trimethylbenzene
Iodomethane			
Acrolein			
Isobutyl Alcohol			
Allyl Chloride			
Chloroprene			
Propionitrile			
Methacrylonitrile			
Acrylonitrile			
Cyclohexane			
Methyl Acetate			
Methylcyclohexane			
1-Chlorohexane			
Dibromofluoromethane (surr.)			
1,2-Dichloroethane-d4 (surr.)			

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 1

EXAMPLE OF VOA RUNLOG PAGE

KATAHDIN ANALYTICAL SERVICES

GCMS-T INSTRUMENT RUNLOG

DATE/TIME OF BFB INJECTION: 7/6/16 0851

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	PREP METHOD 5030	5035	1311	Criteria KAS	DoD	QAPP	pH Paper Lot #: Y/N	ANALYST	PH	COMMENTS
50ng BFB -10	TB174	1	-1	SW8260B52							Y	ERL		WG1BG61
VSTD050 -4	TS100	1	1	T0QG566							Y			
020 -3	01	1	2								Y			
010 -2	02	1	3								Y			
005 -1	03	1	4								Y			
200	04	1	5								N			missing compounds
100 -5	05	1	6								Y			
300 -6	06	1	7								Y			
IWD/LCS	07	1	8								N			MTBE↑
LCS -8/IWD-7(4)	08	1	9								Y			MTBE↑ low
MBLKA	09	1	10								Y			hits PQL
LB -9	10	1	11								Y			no target hits
539595-1G #1	11	1	12			X		X			Y			
-2G #1	12	1	13								Y			
-3G #1	15	1	14								Y			1721✓
-1RAG #2	14	1	15								N			not reviewed
-2RAG #2	15	1	16								N			
-3RAG #2	16	1	17			↓		↓			N			↓
Rinse	17	1	18								N			
Rinse	18	1	19	↓							N	↓		

7/6/16 ERL

STANDARD	CODE
BFB	V0552
CAL STD	V0557 / V0556-05
LCS/MS MIX	V03758
EXTRAS MIX	V0556

STANDARD	CODE
IS MIX	V0585
SS MIX	↓

Circle Methods:

SW846 8260	OLM 04.2
SW846 8260 SIM	OLC 03.2
SW846 8260 SIM (heated purge)	EPA 624
	EPA 524

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QAMS595

0000098

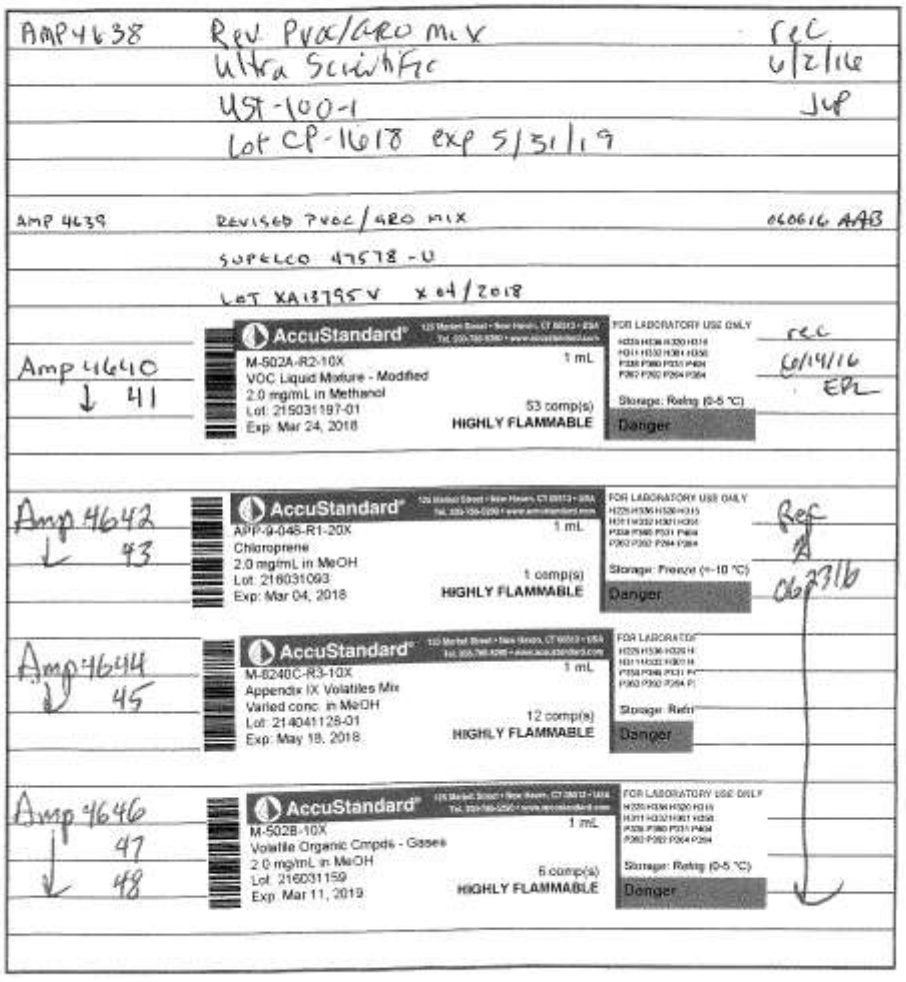
TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
STOCK STANDARDS RECEIVED
GCMS VOLATILES LABORATORY

REVIEWED BY/DATE:



TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS VOA STANDARD PREP LOG BOOK

CODE: V0391	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 524 ES/SS	4-BFB	Amp 4413	2000	250
FINAL CONC (ug/mL): 250	1,2-DCB	4525	↓	250
FINAL VOLUME (mL): 4	Fluorobenzene	↓ 4634	↓	250
PREP DATE: 070716				
EXPIRATION DATE: 073116				
MEOH VOLUME (uL): 4500				
MEOH LOT #: DP303				
INITIALS:				
CODE:	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD:				
FINAL CONC (ug/mL):				
FINAL VOLUME (mL):				
PREP DATE:				
EXPIRATION DATE:				
MEOH VOLUME (uL):				
MEOH LOT #:				
INITIALS:				
CODE:	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD:				
FINAL CONC (ug/mL):				
FINAL VOLUME (mL):				
PREP DATE:				
EXPIRATION DATE:				
MEOH VOLUME (uL):				
MEOH LOT #:				
INITIALS:				

Reviewed by/Date:

VOA-010 - Revision 1 - 04/01/2010 QAMS602 0000021

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

Prepared By: _____ Date: 2/2/06

Approved By:

Department Manager: _____ Date: 2/2/06

Operations Manager: Deborah J. Nadeau Date: 2/2/06

QA Officer: Leslie Dimond Date: 2-2-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Section 7.9 removed surrogate reference. Added information to section 1.4 for headspace vial disposal. Minor changes throughout.	LAD	03/07	03/07
02	Added LCS to definitions and Sect. 8.2 Added N_2 Purging to sect.s 7.1 and 7.2	LAD	01/08	01/08
03	Removed all references to method SW846 3810. updated Figure 1 - Logbook Page.	LAD	06/09	06/09
04	Added independant calibration verification to sections 1.0, 7.4, 7.4.2, 8.2 and Table 1. Section 7.4 and 7.4.1 - corrected tables and formula. Changed CAR to NCR. Updated References.	LAD	09/10	09/10
05	Removed surrogate reference in Section 7.9. Added references to Section 10.	LAD	07/11	07/11

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Sect. 1 and 7 – Removed Quickform reference and added reporting through KIMS. Sect. 8 – Removed annual MDL requirement. Sect. 9 – Added MDL. LOD and LOQ information.	LAD	05/13	05/13

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-336-06, titled “Dissolved Gas Analysis in Water Samples Using GC Headspace Equilibration Technique EPA SOP RSK-175”.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-336-06, titled “Dissolved Gas Analysis in Water Samples Using GC Headspace Equilibration Technique EPA SOP RSK-175”.

Recipient: _____ Date: _____

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of aqueous samples for Methane, Ethane and Ethene by RSK-175, as performed by Katahdin Analytical Services, Inc. including sample preparation, sample analysis, data review, standard preparation and instrument calibration.

1.1 Definitions:

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (LABORATORY REAGENT BLANK): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

CALIBRATION STANDARD (WORKING STANDARD): A standard prepared from the stock standard that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDENT CALIBRATION VERIFICATION (ICV): The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. A reagent water blank is spiked with the ICV Standard and analyzed immediately following a calibration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) of interest and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The LCS is obtained from a source different from the source of the calibration standards.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock standards of certain analytes are added to a sample matrix prior to sample analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. MS/MSD's are spiked with the same standard as the LCS.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation,

generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of Methane Ethane, Ethene by method RSK-175. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability”, current revision.

It is the responsibility of all Katahdin technical personnel involved in the Methane Ethane, Ethene by method RSK-175 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate

personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP. Used headspace vials are disposed of in the "P" waste satellite accumulation area located under GC05.

2.0 SUMMARY OF METHOD

A water sample is collected in a 40 mL VOA bottle using a Teflon faced septum and cap. In the laboratory, a 5 mL aliquot of a sample is injected into a vial and placed onto a headspace autosampler where each sample is shaken and heated prior to injection. The analyte(s) present in the samples will partition between the water and the gas phase according to Henry's Law. The autosampler pressurizes the sample in order to inject 1 mL of headspace onto a gas chromatographic column where the gaseous compounds are separated and detected by a flame ionization detector (FID).

3.0 INTERFERENCES

The sample integrity is compromised if the sample vial contains headspace prior to sample preparation. The presence of headspace in the sample vial is notated in the laboratory narrative.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph: GC Hewlett Packard 5890 series I or II connected to the Turbochrom data system, or equivalent

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

4.2 Headspace Analyzer: Agilent Technologies G1888 Network Headspace Analyzer

4.3 Column: 80/100 mesh Poropak Column 6ft x 1/8”

4.4 Detector: Flame Ionization Detector (FID)

4.5 Data System: A data system which allows the continuous acquisition of data throughout the duration of the chromatographic program must be interfaced to the GC. The data system must be capable of storing and re-integrating chromatographic data and must be capable of determining peak areas using a forced baseline projection. All data editing will be reviewed by the Department Manager or qualified designee before samples are reported.

4.6 Headspace Syringes: various sizes for preparing standards and injecting samples

4.7 5 mL Leur Lock gas-tight syringe with liquid needle

4.8 10 mL headspace vials

4.9 40 mL VOA vials

4.10 Refrigerator for storage of samples

4.11 pH strips (pH 1 – 14 range)

4.12 Tedlar Bags

4.13 Septum cap and crimper

4.14 Brinkmann Pipetter, volume up to 5 mL

5.0 REAGENTS

5.1 Ultra high purity Nitrogen

5.2 Ultra high purity Hydrogen

5.3 Laboratory Reagent Grade Water: Milli-Q, or equivalent

5.4 Certified Gas Standards, Scotty or equivalent

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected into 40 mL VOA vials. The vials have been preserved with 1:1 HCL prior to collection. Care should be taken so there are no air bubbles in the vials.

Samples are stored at 4 (±2) °C until time of analysis. Samples must be analyzed within 14 days of sampling. Unpreserved samples must be analyzed within 7 days of sampling.

7.0 PROCEDURES

7.1 Preparing Samples for Analysis:

Allow samples to warm to room temperature prior to preparation. Inspect all VOA vials for bubbles and notate any bubbles in the logbook.

Purge all headspace vials with nitrogen for approximately ten seconds prior to injecting them with sample. The empty headspace vials are purged and then capped. The nitrogen line is located between the headspace sampler and the GC05 oven.

Using a 5 mL Leur lock syringe, pull up 5 mL of Nitrogen from a Tedlar bag. While inverting a VOA vial push the syringe through the vial septa. Insert a second 5 mL syringe into the VOA vial. By injecting the 5 mL of nitrogen, 5 mL of sample will be displaced into the second syringe. Take the syringe containing the sample aliquot out of the VOA vial and immediately inject the aliquot through the headspace vial septa. The sample is now ready to be loaded onto the autosampler.

If a dilution is required in order to bring the sample within range of the calibration curve, the sample is prepared as above, but less than 5 mL of sample is injected into the vial. An aliquot of laboratory reagent grade water is used to bring the liquid volume to 5 mL. The laboratory reagent grade water is purged with the nitrogen line for approximately ten seconds before capping the headspace vials and adding sample. The amounts of sample and water are based on the factor needed to bring the sample within range of the upper half of the calibration curve. The amount of sample and water are notated in the logbook and the proper dilution factor is applied to the final result.

7.2 Standards Preparation

Using a pipetter, inject 5 mL DI water into a headspace vial, purge with nitrogen for approximately ten seconds and crimp the top. A gas standard is then injected into the vial. The standards are calibrated as µg/mL of water.

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7.3 GC Conditions

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

Nitrogen Carrier:	20 mL/min.
Ultra Zero Air:	400 mL/min.
Hydrogen:	40 mL/min.
Injector Temp.:	200°
Detector Temp.:	250°
Oven Ramp:	40 hold 1 min; 10 degrees/min to 100°
Run time:	7 min
Injection size:	1 mL
Column head pressure:	14 psi

7.4 Calibration

The GC system is calibrated using the external standard calibration procedure. A six-point (5-point minimum) calibration is prepared according to the concentrations listed below. When the calibration curve is run an independent check standard should also be run to validate the curve.

Methane MW=16

Vol of Std inj. (µL)	Std Conc (ppm)	Std. Injected into Vial (µg)	Water Volume (mL)	Water Conc. (µg/mL)
38	1000	0.025	5.0	0.005
200	1000	0.133	5.0	0.027
500	1000	0.333	5.0	0.067
1000	1000	0.665	5.0	0.133
5000	1000	3.326	5.0	0.665
900	10000	5.986	5.0	1.197

Ethene MW=28

Vol of Std inj. (µL)	Std Conc (ppm)	Std. Injected into Vial (µg)	Water Volume (mL)	Water Conc. (µg/mL)
38	1000	0.044	5.0	0.009
200	1000	0.233	5.0	0.047
500	1000	0.582	5.0	0.116
1000	1000	1.164	5.0	0.233
5000	1000	5.819	5.0	1.164
900	10000	10.476	5.0	2.095

Ethane MW=30

Vol of Std inj. (µL)	Std Conc (ppm)	Std. Injected into Vial (µg)	Water Volume (mL)	Water Conc. (µg/mL)
38	1000	0.047	5.0	0.009
200	1000	0.249	5.0	0.050
500	1000	0.624	5.0	0.125
1000	1000	1.247	5.0	0.249
5000	1000	6.236	5.0	1.247
900	10000	11.224	5.0	2.245

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak height or area for each compound. A calibration curve can be prepared in Target using the peak height or area against the concentration of the standard. An average calibration applying a first order polynomial equation is used to prepare the curve.

7.4.1 Calculating the concentration of the calibration standard (x)

$$\text{µg std. injected into vial} = \frac{(\text{ppmv of std.})(\text{MW of gas})(\text{mL injected})}{24055}$$

$$\text{conc.} = \frac{\text{µg std. injected into vial}}{\text{amount of water in vial (mL)}}$$

7.4.2 An Independent Calibration Verification Standard (ICV) is analyzed immediately after calibration, before any samples are analyzed.

7.5 Retention Time Study

Three injections are made of all the analytes throughout the course of a 72-hour period.
A major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms. Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

7.6 Sample Analysis

Each vial may only be analyzed once. If a second analysis is required, the sample must be re-prepped. The samples and standards are loaded onto the autosampler, which heats and shakes the vials. The autosampler then pressurizes the sample to fill the 1 mL sample loop. The 1 mL headspace sample is then injected into the GC instrument.

Samples are analyzed in a set referred to as an analytical sequence. The sequence begins with instrument calibration as listed in section 7.4 followed by sample aliquots interspersed with mid-concentration calibration standards.

Before any samples are analyzed the instrument must be calibrated by analyzing a five-point (minimum) calibration or a mid-concentration standard (calibration verification standard). If a CV is run, the calculated concentration must not exceed a difference of ± 30%. Each sample analysis must be bracketed with an acceptable initial calibration and a closing CV, or an opening CV and a closing CV. The calibration standard must also be injected at intervals of not less than once every twenty samples (or every 12 hours), whichever is more frequent, and at the end of the analysis sequence.

If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. All samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e. >30%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the analyses for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits was detected in a sample analysis, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 30% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

Absolute retention time windows are established using the mid-point of the window of that day if after analyzing the mid-point it is determined that one or more of the analytes fall outside of the previously established absolute retention time window. The daily retention time window equals the mid-point ±three times the standard deviations.

The identification of methane, ethane and ethene is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows

established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the daily retention time window.

If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

If the amount recovered is not detectable or below the PQL, then the compound is not considered to be present in the sample and is reported as <PQL.

When a GC system is determined to be out of control because either a CV can not pass or a six-point calibration does not meet the correlation coefficient criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the FID or an electronic board, this information is written in the instrument maintenance logbook.

7.7 Calculations

The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibrated method.

Concentration (µg/L) = $[(C)\ (0.005L)/(V_s)]\ (1000)$

Where: C = Concentration calculated by Target in µg/mL
V_s = Volume of sample purged in L

7.8 Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed in Target Review. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or reextracted.

These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.

- Chromatography: cleanups, manual integration.

- Target compound detection: quantitation and false positives.

- The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.10.

7.9 Chromatography

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

In Target Review, each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), manual integration is performed in Target Review. An "M" qualifier will automatically be printed on the quantitation report summary indicating that a manual integration was performed. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration," current revision.

7.9.1 Target Compound Detection

The chromatogram is evaluated to determine if a target analyte is indicated. The concentration of the analyte(s) is then evaluated to determine if it is above the PQL and within the calibration range.

7.10 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

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8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The Department Manager, Laboratory Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

Every instance of noncompliant method quality control requires the generation of a Nonconformance Report (NCR) describing the problem, suspected cause and final resolution. A NCR must be initiated as soon as possible.

8.1 Continuing Calibration Verification (CV)

A mid-level concentration standard is analyzed daily prior to sample analysis. The calibration standard must also be injected at intervals of not less than once every twenty samples (or every 12 hours), whichever is more frequent, and at the end of the analysis sequence. The acceptance criterion is ±30% of the expected value. If response of a compound, in the opening CV, fails to met the criterion, the system is checked, the standard reprepped and analyzed. In the event the criterion cannot be met, the instrument is recalibrated.

8.2 Independent Calibration Verification (ICV)

An ICV is a mid-level concentration standard using a source different from the source of the calibration standards. This can include a different lot from the same manufacturer. An ICV is analyzed immediately following a curve. The acceptance criterion is ±30% of the expected value. If the ICV fails to meet this criterion, the

system is checked, and another ICV is analyzed. In the event the criterion cannot be met, the instrument is recalibrated.

8.3 Laboratory Control Sample (LCS)

An LCS is a mid-level concentration standard using a source different from the source of the calibration standards. This can include a different lot from the same manufacturer. An LCS is analyzed prior to sample analysis. The acceptance criterion is ±30% of the expected value. If the compound recovery fails to meet this criterion, the system is checked, and another LCS is prepped and analyzed. In the event the criterion cannot be met, the instrument is recalibrated.

8.4 Laboratory Blank

The Laboratory Blank is prepared by injecting 5 mL of DI water into a 10 mL headspace vial. A Laboratory Blank is analyzed between analysis of standards and project samples. If analytes are detected above the detection limit, the blank is reprepped and analyzed. If analytes are still detected above the detection limit, the possibility exists that all the vials in the batch contain contamination. In this case all samples and QC are reprepped in new vials.

8.5 Sample Duplicates

Sample duplicates are analyzed as required for certain clients. The duplicate is prepared using a second VOA sample using the procedures in section 7.1.

8.6 Detection Limits

An Method Detection Limit (MDL) study is preformed using a minimum of seven replicates at 1-2 times the Practical Quantitation Limit (PQL) or Reporting Limit (RL) described in 40 CFR Pt. 136 App. B. The MDL must be less than or equal to the detection limit.

Compound	PQL or RL (µg/L)
Methane	10
Ethane	10
Ethene	10

8.7 Matrix Spikes and Matrix Spike Duplicates (MS/MSD)

For projects requiring MS/MSD sets, aliquots of the sample are prepared using the procedures in section 7.1. The MS/MSD's are then spiked in the same fashion as the LCS. After analysis, the original sample amount is subtracted out and the % recovery is calculated. The acceptance criterion for MS/MSD sets are 70-130%

recovery and 30% RPD. If the criteria are not met, the data is flagged and the incident is narrated. Since samples are analyzed using an autosampler, it is not possible to know in advance the concentration of the sample. Consequently, the concentration of analytes in the unspiked analysis may be greater than four times the concentration of the added spike, making the spike amount insignificant to the original concentration. In these situations, recoveries and RPD may not meet the acceptance criterion. In addition, as MS/MSD's are typically taken from separate vials, sample heterogeneity may contribute to failed criteria.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Method RSK-175 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Analysis of Dissolved Methane, Ethane and Ethylene in Ground Water by a Standard Gas Chromatographic Technique", EPA SOP RSK-175, Revision No. 0, 8/11/94

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

Katahdin SOP CA-101, “Equipment Maintenance and Troubleshooting,” current revision.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Current Version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Katahdin SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications,” current revision.

LIST OF TABLES AND FIGURES

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

TABLE 1

SUMMARY OF CALIBRATION AND QC PROCEDURES

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
ICAL	Initial calibration prior to sample analysis	RSD ≤ 30%	Investigate and repeat initial calibration
ICV	Immediately following initial calibration	Recovery must be between 70% and 130%	Investigate; reprep. Repeat initial calibration if criteria cannot be met.
CV	If initial calibration analyzed, daily and after 20 samples, and at end of sequence.	%D for all analytes within 30%	(1) Evaluate the samples: (2) If the %RPD >30% and sample results are < PQL, narrate. (3) If %RPD >30% and is likely a result of matrix interference, narrate. (4) Otherwise, reanalyze all samples after last acceptable CV.
LCS	One LCS per 20 samples	Recovery must be between 70% and 130%	(1) Evaluate the samples and associated QC. (2) If an MS/MSD was performed and acceptable, narrate. (3) If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep.
Method Blank	One per batch of 20 or fewer samples	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report samples results which are < PQL >10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
Matrix Spike/Matrix Spike Duplicate	One MS/MSD as requested by clients.	Recovery must be between 70% and 130%, RPD ≤30.	(1) Evaluate the samples and associated QC. (2) If the LCS is acceptable, narrate. (3) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample Duplicate	If requested by the client	%RPD of duplicate must be less than 30%.	(1) Check calculations for errors (2) Evaluate QC
Demonstration of capability - four replicate analyses of a QC check sample	One time per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Investigate; reprep
MDL and/or LOD/LOQ Verification	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications," current revision.		

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

TABLE 2

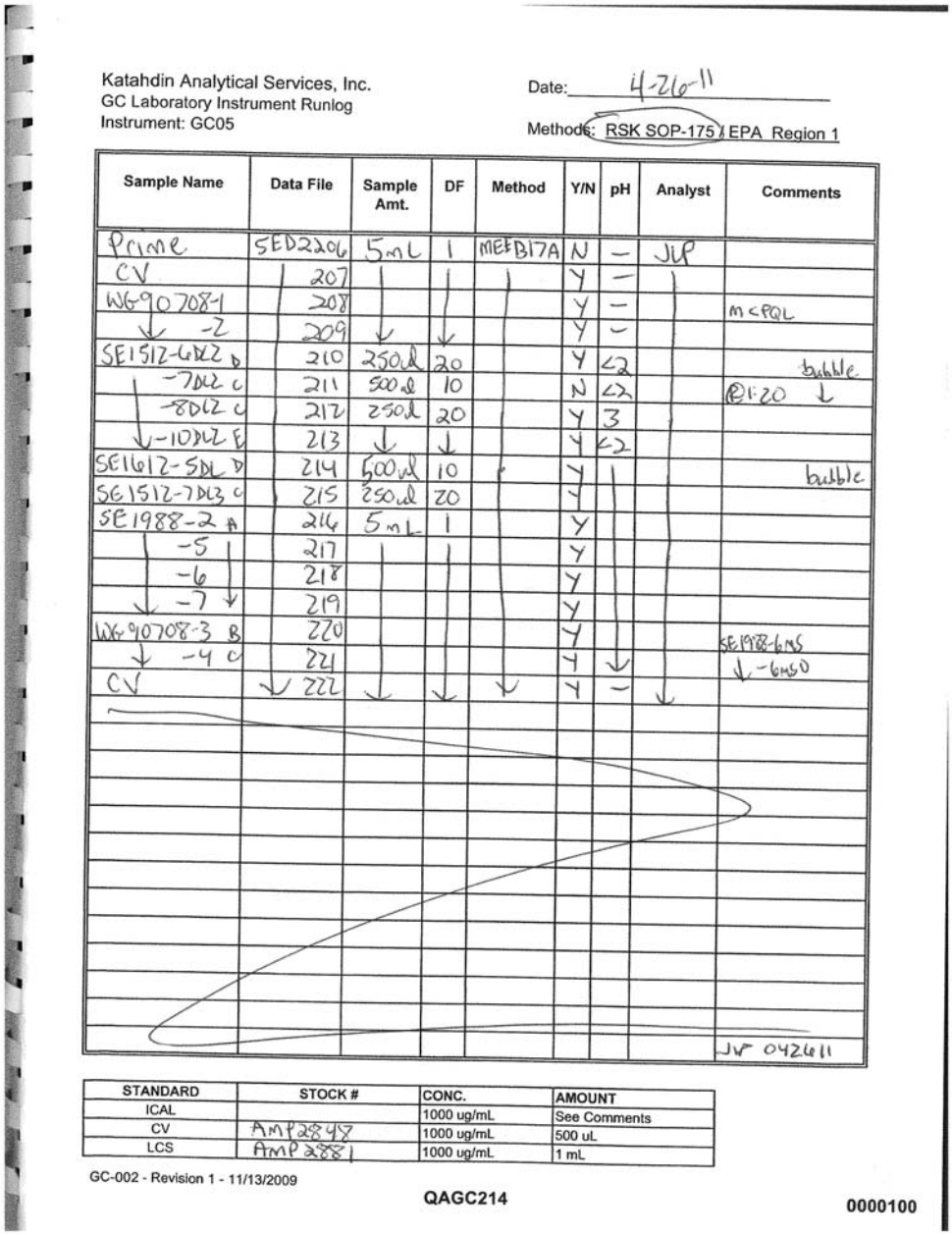
SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-336-06	Method: EPA SOP RSK-175
Apparatus/Materials		
Reagents		
Sample preservation/ handling	(1) Collect sample in 40 mL VOA vial. (2) HCL added in field; hold time is 14 days, un-preserved is 7 days.	(1) Collect sample in 60 mL crimp top vial. (2) HCL is added in field; hold time is 14 days.
Procedures	(1) 5 mL of sample is displaced with 5 mL of nitrogen and transferred to a capped autosampler vial. Headspace is then generated in the autosampler vial. (2) Prior to injection, autosampler shakes sample for 15 min while heating to 40°C. (3) Autosampler pressurizes sample to fill 1 mL loop with headspace sample. (4) Calibration is obtained by spiking headspace samples with gas phase analyte and analyzing using the same procedure as the samples. Quantitation of samples is directly obtained using the calibration curve that relates µg analyte/mL water sample to peak area. (5) ICAL using average response factor	(1) Headspace is generated in 60 mL vials by displacing volume of liquid with helium. The amount of liquid should be 10% of sample volume in bottle, up to 10mL. (2) Sample is shaken 5 min to equilibrate analyte between headspace and liquid phase. (3) Syringe injections of 300 µL headspace into GC. (4) Direct injections of gas phase standards are used to obtain a calibration curve. Henry's law is used to calculate mg of gas per L of water. Calculation requires recording total volume of serum bottle and headspace, and sample temperature. (5) ICAL using linear regression
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL	See Section 9 of this SOP.	No information.

TITLE: DISSOLVED GAS ANALYSIS IN WATER SAMPLES USING GC HEADSPACE EQUILIBRATION TECHNIQUE EPA SOP RSK-175

FIGURE 1

EXAMPLE OF RUNLOG



Katahdin Analytical Services, Inc.
GC Laboratory Instrument Runlog
Instrument: GC05

Date: 4-26-11

Methods: RSK SOP-175 / EPA Region 1

Sample Name	Data File	Sample Amt.	DF	Method	Y/N	pH	Analyst	Comments
Prime	SED2206	5mL	1	MEEB17A	N	—	JLP	
CV	207				Y	—		
WG907087-1	208				Y	—		m<PQL
-2	209				Y	—		
SE1512-6DL2 D	210	250µl	20		Y	<2		bubble
-7DL2 C	211	500µl	10		N	<2		@1:20
-8DL2 C	212	250µl	20		Y	3		
-10DL2 E	213				Y	<2		
SE1612-5DL D	214	500µl	10		Y			bubble
SE1512-7DL3 C	215	250µl	20		Y			
SE1988-2 A	216	5mL	1		Y			
-5	217				Y			
-6	218				Y			
-7	219				Y			
WG907087-3 B	220				Y			SE1988-6MS
-4 C	221				Y			-6MSD
CV	222				Y	—		

JV 042611

STANDARD	STOCK #	CONC.	AMOUNT
ICAL		1000 ug/mL	See Comments
CV	AMP28487	1000 ug/mL	500 uL
LCS	AMP28881	1000 ug/mL	1 mL

GC-002 - Revision 1 - 11/13/2009

QAGC214

0000100

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Carolyn Byrne

Review Date: 3/11/14

SOP Number: CA-336-06

SOP Title: Dissolved Gas Analysis in Water Samples Using GC Headspace Equilibration Tech. ~~Using~~ with SOP LSE-175

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 8-28-14

QAO Signature: Leslie Dimond **Date:** 082814

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Jessica Spearn-Wilder

Review Date: 6-30-15

SOP Number: CA-336.06

SOP Title: EPA SOP RSK-175 MEE

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 6-30-15

QAO Signature: Jessie Dimond **Date:** 07.01.15

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Jessica Spearin-Wildes

Review Date: 6.9.16

SOP Number: CA-336-06

SOP Title: EPA SOP RSK175 MEE

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 6-9-16

QAO Signature: Leslie Dimond **Date:** 06.09.16

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Buxton Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Duncan F. Heffron Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DH	1·22·01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DH	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09
05	Added references. Updated Figure 2 and 3 with correct spike information. Added CA-108 reference for subsampling information.	LAD	04/10	04/10

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated Figures 2 and 3. Changed KAS, INC. to KAS.	LAD	06/15	06/15
07	Sect. 1 - Replaced beaker with digestion vessel. Sect. 7.5 & 7.9 - Added calibrated pipet.	LAD	06/16	06/16

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-604-07**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-07**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever digestion vessels are being heated in the hood. Use caution when handling hot digestion vessels.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids

and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95^{o}C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for information on subsampling.

7.0 PROCEDURES

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet. With a permamament marker, make sample labels and attach to the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter or calibrated pipet, to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

NOTE: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample and use a repipetter or calibrated pipet to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) the sample bottle ID, reagent lot numbers, spiking information, initial and final

volumes, hot plate ID and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 3010A.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.2, 10/25/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

LIST OF TABLES AND FIGURES

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-07	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume. 2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
2. Label digestion vessels with sample numbers.
3. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
4. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
5. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
6. Cover with a ribbed watch glass.
7. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
8. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
11. Evaporate to 10 - 15 mL.
12. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
13. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
14. Dilute to appropriate final volume with reagent water.
15. Cap sample container and shake gently to mix.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 1

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — **Metals Preparation Benchsheet**

Reagent Information:
JT Baker HNO3: h14024 JT Baker HCL: 113907 KMG H2O2: N/A **Method:** 3010

LCS / Spike — **LCS/Spiking Information:**

☑	☑	LV. CLPP-SPK-1 (ID/Vol):	MS1304	/ 0.05	mL
☑	☑	CLPP-SPK-INT1 (ID/Vol):	MU1203	/ 0.5	mL
☑	☑	CLPP-SPK-INT2 (ID/Vol):	MU 0204	/ 0.5	mL
☑	☑	Uranium Spike (ID/Vol):	MS1269	/ 0.005	mL
☐	☐	CLPP-SPK-4 (ID/Vol):	N/A	/	mL

Hot Plate/Block ID : A
Start Time/Temp.: 930 / 95 °C
End Time/Temp.: 1500 / 55 °C
Thermometer ID/Pos.: AIC 4 / 1:4
Fisher Filter Paper: W1167032S 2

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWAB01ICW0	AB01ICW0	0.05	L	0.05	L	AQ	IC	AJB	02/01/2010	N/A	N/A	N/A	N/A		
PBWAB01ICW0	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010	N/A	N/A	N/A	N/A		
SD0405-001	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						H
SD0405-001P	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-001S	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-002	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-003	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-004	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-005	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0422-001	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						B
SD0423-001	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						A
SD0429-001	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						D
SD0429-002	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-001	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-002	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-003	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-004	AB01ICW0		L		L	AQ	IC	AJB	02/01/2010						

ASD 2-1-10

Digestion performed by: AJB On: 2-1-10 **Page:** AB003 **Revision:** 00

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	1.0
	1000 mg/L As	High Purity Standards	1.0
	1000 mg/L Pb	High Purity Standards	1.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	1.0
	10,000 mg/L K	High Purity Standards	10.0
	10,000 mg/L Na	High Purity Standards	7.5
	10,000 mg/L Mg	High Purity Standards	5.0
	10,000 mg/L Ca	High Purity Standards	2.5
	1000 mg/L Tl	High Purity Standards	1.0
CLPP-SPK-INT2	1000mg/L Sr	High Purity Standards	5.0
	1000mg/L Sn	High Purity Standards	5.0
	10,000mg/L Si	High Purity Standards	1.0
	1000mg/L B	High Purity Standards	5.0
	1000mg/L Li	High Purity Standards	5.0
	1000mg/L Ti	High Purity Standards	5.0
	1000mg/L Mo	High Purity Standards	1.0
	1000mg/L U	High Purity Standards	1.0
	1000mg/L W	High Purity Standards	1.0

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION		
	CLPP-SPK-1	CLPP-SPK-INT1	CLPP-SPK-INT2
Aluminum	2000		
Antimony		10	
Arsenic		10	
Barium	2000		
Beryllium	50		
Boron			50
Cadmium		25	
Calcium		250	
Chromium	200		
Cobalt	500		
Copper	250		
Iron	1000		
Lead		10	
Magnesium		500	
Manganese	500		
Molybdenum			10
Nickel	500		
Potassium		1000	
Selenium		10	
Silicon			100
Silver	50		
Sodium		750	
Strontium			50
Thallium		10	
Tin			50
Titanium			50
Uranium			10
Vanadium	500		
Zinc	500		
Lithium			50
Tungsten			10

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Prepared By: George Brewer Date: 7/98

Approved By:

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: Jd C. Burton Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1.23.01

General Manager: Duncan F. Hodgson Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DH	1.23.01	1/23/01
02 6010B	Calibration begins with analysis of S0 (cal. blank) followed by S1 (Mixed Cal. Std.) Changes to section 7.5 and Table 8 to reflect this. Made changes to element concs. in Tables 3,4,5,6 to reflect current practices.	DH	10.21.02	10.21.02
03 6010B	Added MN-IEC to standards run. Changed frequency of LRS. Changed concentration of HNO_3 in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	Updated ICV, CCV, ICB, PQL Chk std. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06
05	Updated Tables 3,4,5,6 and 7 with current Standard concentrations and prep. Updated Table 1 with current practices including NAVY award findings. Updated Sections 2, 7.2, 7.6 and Table 1 with new ICP information. Updated Table 8 with current sequence requirements.	LAD	07/07	07/07

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added hardness definition and calculation (APP. 1)	LAD	09/07	09/07
07	Updated Summary to reflect new ICP functions. Removed ICP set-up Updated tables to reflect changes in Standard Concentrations and Preparation	LAD	11/08	11/08
08	Updates to Sections 8 and 10, Tables 1 and 2 to reflect changes from 6010B to 6010C. Added LLQC information and criteria to Sect. 8 and Table 1. Added criteria to analyze PQL standard at the beginning and END of each run.	LAD	02/09	02/09
09	Updated Sections 8, 9, 10 and Table 1 for compliance with DoD QSM version 4.1.	LAD	08/09	08/09
10	Added Table 2 - DoD QSM Ver. 4.1 QC Requirements. Minor correction to Table 1.	LAD	04/10	04/10
11	Added yttrium criteria to section 7 and Table 1.	LAD	06/10	06/10
12	Revised Tables 4 → 8 with the following information: -Add palladium and gold; removed tungsten and uranium; removed Stock Standard ~~ICP-CICV~~ 2007ICS-1; changed stock standard ICP-CICV-3 to CL-CAL-3. Added references to section 10. LAD 092211	LAD	09/11	09/11
13	The changes above had not been finalized in SOP-12. Sect. 9 - Added MDL, LOD and LOQ information. Added Attachment 2 - Analysis of Palladium by SW846 6010	LAD	04/12	04/12

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 9 & Table 1 - Fixed typos.	LAD	05/13	05/13
15	Sect. 10 - Updated references. Added Table 3 - DoDQSM 5.0 QC Requirements - Renumbered rest of Tables. Updated Tables (6→8). Changed KAS INC to KAS LLC.	LAD	12/14	12/14
16	Sect. 5 & 7 - Corrected Table references. Tables 5, 6, 7 & 8 - Updated Standard, Concentrations & Sources. Changed KAS LLC to KAS	LAD	05/16	05/16
17	Sect. 1 and 6 - Added Tissue matrix	LAD	07/16	07/16

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-608-15**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

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TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, LLC personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010 (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, biological tissue and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010 require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

1.1 Definitions

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

CRI - Contract Required detection limit sample for ICP - A low concentration standard used to verify calibration accuracy near the low end of the calibration range.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution

ICSA contains only interferents (Al, Ca, Fe, and Mg) at high concentrations (200 to 500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document

their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual l and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial and axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating and the intensities of the emitted lines are monitored by a solid state charge injection device (CID) camera system. Photocurrents from the CID camera system are measured by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of

the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction. For more information refer to the current revision of Katahdin SOP CA-632, “Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer”.

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS AND STANDARDS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. This solution is also used to flush the system between standards and samples. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.

5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 5 and 6 for a listing of stock standards required, and to Table 9 for element concentrations in stock standards.

5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 6 for a listing of intermediate standards required and for preparation instructions. Refer to Table 8 for element concentrations in intermediate standards.

5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 5 for a listing of working standards and for preparation instructions. Refer to Table 7 for element concentrations in working standards.

5.8 5 mg/L yttrium internal standard solution – add 0.5 mL 10000 mg/L yttrium stock standard to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container[1]	Volume / Weight	Preservation / Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months
Tissue	P, G	25 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Begin by following the startup and calibration instructions provided in the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer"

7.2 Analysis must proceed in the sequence described in Table 10 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with the analysis of a calibration blank solution (S0) followed by analysis of a multi-element calibration standard (S1 in Table 5) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.3 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Tables 1 through 3 for additional information.

7.4 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Tables 1 through 3 for additional information.

7.5 Interference check standard solutions (ICSA and ICSAB) must be analyzed at the beginning, end, and at periodic intervals (4-6 hours, 30-40 analytical samples) throughout the sample run to verify the accuracy of the IEC factors. Refer to Section 8 and Tables 1 through 3 for additional information.

7.6 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Tables 1 through 3 for additional information.

7.7 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.

7.8 All samples that exceed the linear dynamic range must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the laboratory for lead analysis, and the measured aluminum concentration of that sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear dynamic range and the lead result must be reported from that dilution analysis.

7.9 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

7.10 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization. For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium. The yttrium recovery must be within ± 20% of the counts of the initial calibration blank. If the recovery is outside of this, the sample must be diluted and reanalyzed.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and

project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed. Tables 2 and 3 list the QC Check, minimum frequencies, acceptance criteria, corrective actions, flagging criteria and additional comments for work analyzed in accordance with DoD QSM versions 4.2 and 5.0.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Limits of Quantitation (LOQ) are used when evaluating data using DoD QSM. The LOQ must be above the LOD.

8.5 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.10) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

8.6 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration differs by no more than 10% from the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analyses of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified **every six months** or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.7 The alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects. These curves may be used for quantitation of samples if the effective range is checked and if the second order curve fit has a correlation coefficient of 0.998 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

ANALYTICAL RUN QC SAMPLES

8.8 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

No results may be accepted for failing elements if DoD QSM acceptance criteria are being used.

8.9 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements

may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL (less than reporting limit for DoD QSM). Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.10 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB or ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

If DoD QSM acceptance criteria are being used, the absolute values of results of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed.

8.11 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within $\pm$ PQL if the PQL is greater than 0.01 mg/L, within $\pm$ 2xPQL if the PQL is less than or equal to 0.01 mg/L. If DoD QSM acceptance criteria are being used, the absolute value of unspiked elements must be less than the LOD. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.12 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 70-130% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run,

unless the PQL Check Standard recovery is greater than 130% and the samples results are less than the PQL.

If DoD QSM acceptance criteria are being used, recoveries must fall between 80-120%. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run.

PREPARATION BATCH QC SAMPLES

8.13 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.

8.14 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.15 A laboratory control sample (LCS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, sample results less than the PQL may be reported.

If DoD QSM 4.2 acceptance criteria are being used, recovery for solid matrix samples must fall between 80% to 120% except for Ag, which must fall between 75% and 120%. If DoD QSM 5.0 acceptance criteria are being used, recovery for water and solid matrix samples must fall between the limits stated in Tables 3 & 4 of the QSM. Results may not be reported without a valid LCS and will be qualified and explained if reanalysis cannot be performed.

SAMPLE MATRIX QC SAMPLES

8.16 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = sample result
D_2= duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

For DoD QSM samples a Post-digestion Spike (PDS) addition must be performed if the serial dilution is not within acceptance criteria.

8.16 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in

all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6010C.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.2, 10/25/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, current revision.

List of Tables & Figures

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ® ≥ 0.998	Recalibrate
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct 3) DoD: No samples may be run until calibration is verified
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL.	1) Do not use results if ≥ PQL and 10x< CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the CCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Practical Quantitation Level Check Standard (PQL) (LLCCV)	At beginning and end of run.	Recovery within ± 30% of true value.	1) Do not use results for failing elements unless the LLCCV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Interference Check Solution A (ICSA)	At beginning and end of run.	For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	At beginning and end of run.	Recovery of each analyte within ± 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples. 3) DoD: Flag specific analytes if samples cannot be reanalyzed.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	1) Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added. RPD ≤20% for duplicate spikes and sample duplicates.	1) Flag results.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010 (cont.)	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result. Flag result or dilute and reanalyzed sample to eliminate interference	Perform post digestion spike addition (PDS)
	Post-Digestion Spike Sample (A)	When dilution test fails or analyte concentration in all samples <50x LOD	Recovery within ± 25%.	Run associated samples by method of standard addition or flag results.
	Internal Standard	Every sample	± 20% (compared to the initial calibration blank)	Dilute sample and reanalyze.
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL > 2-3 * the IDL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Re-evaluate PQLs
	Linear Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.
	Limit of Detection (LOD) Determination	Quarterly	LOD = 1-4X MDL	Repeat LOD Determination
	Limit of Quantification (LOQ) Determination	Quarterly	LOQ > LOD	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months.	Within ± 10% of true value.	NA.	NA.	
Initial calibration (ICAL) for all analytes ICP: minimum one high standard and a calibration blank	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r ≥ 0.995.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	ICP: within ± 10% of true value; GFAA: within ± 20% of true value; CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	After every 10 field samples and at the end of the analysis sequence.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For negative blanks, absolute value < LOD.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS)	At the beginning of an analytical run.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported	One per preparatory batch.	Water and Soil: Recovery must be within + 20% of the true value	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix	For matrix evaluation, recovery must be within +/- 20% of the true value.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation, recovery must be within +/- 20% of the true value. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution test	One per preparatory batch.	If sample concentrations > 50 x LOQ, then the five-fold dilution must agree within ± 10% of the original measurement.	Perform post-digestion spike (PDS) addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post-digestion spike (PDS) addition	When dilution test fails or analyte concentration in all samples < 50 x LOD.	Recovery within 75-125%.	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 3

DoD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples within the calibration range, or re-establish/ verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the high calibration range without an established/passing high-level check standard.
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low-level ICV)	Daily.	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard (LLICV). Low-level calibration check standard should be less than or equal to the LOQ.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 3

DoD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Flagging is not appropriate.	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike(MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD)	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J- flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.

TABLE 3

DoD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Dilution Test	One per preparatory batch if MS or MSD fails	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J- flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.
Post-Digestion Spike (PDS) Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible)	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria applies for samples with concentrations <50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA	NA	NA	Document use of MSA in the case narrative.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 4

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-608-17	Method 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: ± PQL	Acceptance criteria stated in 6010: less than 10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 5

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1)	ICP- intermediate Standard	Lab Prepared (see Table 6)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	Calibration Standard 3	Claritas PPT	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	High Purity Standards	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn, Au	Inorganic Ventures	0.04
Interference Check Sample A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Sample AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 6)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 6)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 6)	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 6

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
PQL-INT	1000 mg/L Li, Sn, Au	High Purity Standards or Inorganic Ventures	1.0 each
	10000 mg/L K, Na	High Purity Standards or Inorganic Ventures	1.0 each
	1000 mg/l B	High Purity Standards	0.50
	1000 mg/l Zn	High Purity Standards	0.20
	1000 mg/L Cu	High Purity Standards	0.25
	10000 mg/L Si	High Purity Standards	0.20
	1000 mg/L Ti, Tl	High Purity Standards	0.15 each
	1000 mg/L Se, Mo, Co, Ni, Ag, Sr, V, Cr	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba, Be, Cd, Mn, Pb	High Purity Standards	0.05 each
	10000 mg/L Fe, Ca, Mg	High Purity Standards	0.1 each
ICSAB-INT	10000 mg/L K,Na	High Purity Standards or Inorganic Ventures	4.0 each
	10000 mg/L B, Li, Mo,Sr,Sn,Ti, Au	High Purity Standards	1.0 each
	10000 mg/L Si	High Purity Standards	0.40
ICP-INT STD (Intermediate)	10000 mg/L Si	High Purity Standards	2.5
	10000 mg/L Ca, Mg, Fe, Al, Na	High Purity Standards	2.4
	10000 mg/L K	High Purity Standards	1.5
	1000 mg/L Au, Li, Sn. Sr	High Purity Standards	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 7

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

	CONCENTRATION IN SOLUTION, mg/L								
Element	**STD1**	**ICV**	**PQL**	**ICSA**	**ICSAB**	**CCV**	**AL_IEC**	**FE_IEC**	**MN_IEC**
Aluminum	25	10	0.3	500	500	12.5	500		
Antimony	1	0.4	0.008		0.6	0.5			
Arsenic	1	0.4	0.008		0.1	0.5			
Barium	1	0.4	0.005		0.5	0.5			
Beryllium	1	0.4	0.005		0.5	0.5			
Boron	1	0.4	0.05		0.5	0.5			
Cadmium	1	0.4	0.005		1.0	0.5			
Calcium	25	10	0.10	500	500	12.5			
Chromium	1	0.4	0.01		0.5	0.5			
Cobalt	1	0.4	0.01		0.5	0.5			
Copper	1	0.4	0.025		0.5	0.5			
Iron	25	10	0.1	200	200	12.5		200	
Lead	1	0.4	0.005		0.05	0.5			
Lithium	1	0.4	0.1		0.5	0.5			
Magnesium	25	10	0.10	500	500	12.5			
Manganese	1	0.4	0.005		0.5	0.5			10
Molybdenum	1	0.4	0.01		0.5	0.5			
Nickel	1	0.4	0.01		0.5	0.5			
Potassium	25	13.6	1		20	12.5			
Selenium	1	0.4	0.01		0.05	0.5			
Silicon	1	0.4	0.2		2	0.5			
Silver	1	0.4	0.01		0.2	0.5			
Sodium	25	10	1		20	12.5			
Strontium	1	0.4	0.01		0.5	0.5			
Thallium	1	0.4	0.015		0.1	0.5			
Tin	1	0.4	0.1		0.5	0.5			
Titanium	1	0.4	0.015		0.5	0.5			
Vanadium	1	0.4	0.01		0.5	0.5			
Zinc	1	0.4	0.02		1.0	0.5			
Gold	1	.04	0.1		0.5	0.5			

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 8

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L		
	ICP Intermed STD	PQL-INT	ICSAB-INT
Aluminum	240	30	
Antimony		0.8	
Arsenic		0.8	
Barium		0.5	
Beryllium		0.5	
Boron		5	10
Cadmium		0.5	
Calcium	240	10	
Chromium		1.0	
Cobalt		1.0	
Copper		2.5	
Iron	240	10	
Lead		0.5	
Lithium	10	10	10
Magnesium	240	10	
Manganese		0.5	
Molybdenum		1.0	10
Nickel		1.0	
Potassium	150	100	400
Selenium		1.0	
Silicon	250	20	40
Silver		1.0	
Sodium	240	100	400
Strontium	10	1.0	10
Thallium		1.5	
Tin	10	10	10
Titanium		1.5	10
Vanadium		1.0	
Zinc		2.0	
Gold	10	10	10

TABLE 9

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	IV-28	QCS-26	CLPP-ICS-A	CLPP-ICS-B4	CL-CAL-3
Aluminum	100	100	5000		
Antimony	100	100		60	
Arsenic	100	100		10	
Barium	100	100		50	
Beryllium	100	100		50	
Boron	100	100			
Cadmium	100	100		100	
Calcium	100	100	5000		1000
Chromium	100	100		50	
Cobalt	100	100		50	
Copper	100	100		50	
Iron	100	100	2000		1000
Lead	100	100		5	
Lithium	100				
Magnesium	100	100	5000		1000
Manganese	100	100		50	
Molybdenum	100	100			
Nickel	100	100		100	
Potassium	1000	1000			1000
Selenium	100	100		5	
Silicon	50	50			
Silver	100	100		20	
Sodium	100	100			1000
Strontium	100				
Thallium	100	100		10	
Tin					
Titanium	100	100			
Vanadium	100	100		50	
Zinc	100	100		100	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 10

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10-19	Analyze up to 10 samples	
20	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

ATTACHMENT 2

ANALYSIS OF PALLADIUM BY SW846 6010

Palladium may be analyzed by EPA Method SW846 6010C following the method outlined in this SOP. However, due to significant spectral interferences caused by addition of palladium to the calibration and check standards used in this method, palladium is added to aliquots of the regular standards as needed for analysis. Two stock standards (1000 mg/L) are currently kept for palladium analysis. One is purchased from High Purity Standards and is used for calibration, PQL, ICSAB, and CCV. The other is purchased from Inorganic Ventures and is used as the independent check standard (ICV). Analysts should add palladium stock to the regular standards according to the table below:

Name of Working Standard	Volume of Standard Aliquot (mL)	Volume of Palladium Stock Added (mL)	Concentration of Palladium (mg/L)	Source of Palladium Stock
Calibration Std.	50	0.05	1.0	High Purity
ICV	50	0.02	0.4	Inorganic Ventures
PQL	50	0.005	0.1	High Purity
ICSAB	50	0.025	0.5	High Purity
CCV	50	0.025	0.5	High Purity

Prior to starting the run, a palladium-only standard should be analyzed along with the iron and aluminum standards to evaluate interelement correction factors as outlined in Katahdin SOP CA-632, Section 7.1.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Joel C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1·29·01

General Manager: Dennis P. Hoffman Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	DH	1·29·01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC limits. Minor changes throughout	LAD	02·16·05	02·16·05
02	Updated Fig. 1 - new prep logbook page	LAD	04/08	04/08
03	Updated Figure 1 - Example of a mercury Preparation Logbook page.	LAD	03/09	03/09
04	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	DH	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
05	Added Table 2 - DoDQSM version 4.1 QC Requirements.	LAD	04/10	04/10
06	Sect. 4.6 - Changed thermometer type. Sect. 7.3 - Changed type of marker used. Table 1 - Added PQL standard corrective action. Table 2 - added comments for calibration blank. Sect. 9 - Added MDL, LOD and LOQ information	LAD	05/11	05/11
07	Sect. 7 - Calibration prep from digesting all to digesting high STD. and diluting down. Added serial dilution and PDS to sect. 8. Added more MDL, LOD & LOQ information to Sect. 9. Updated and added references to Sect. 10	LAD	04/12	04/12
08	DoDQSM 5.0 Reperences added. Sect. 7.4 and Table 3 - Updated Calibration Standard prep - removing digesting all Standards. Added to digest high point	LAD	06/14	06/14

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

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TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.
ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this

method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 40 mL VOA vials, for use as digestion vessels.

4.2 250 mL Pyrex media bottles with plastic screw caps, for use in digesting calibration standards.

4.3 Water bath capable of maintaining a constant temperature of 95^{o} C.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.6 Battery powered Traceable Pocket-Size Thermometer from Fisher Scientific, NIST-traceable, covering the range from -50^{o} to 750^{o} C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity

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4.8 CETAC M-6100 automated mercury analyzer and associated peripherals and parts

4.9 Disposable graduated dose cups, 30 mL capacity

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer" for additional required materials.

5.0 REAGENTS

5.1 Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water

5.2 Concentrated sulfuric acid, trace metals grade

5.3 Concentrated nitric acid, trace metals grade

5.4 Concentrated hydrochloric acid, trace metal grade

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Potassium persulfate solution, 5% w/v: Dissolve 50g of potassium permanganate in 1L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.7 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 1 L.

5.8 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh monthly ,and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly, and disposed of appropriately after use.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO_3 to pH < 2	28 days

[1] P = polyethylene or G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in 250 mL Pyrex media bottles. Large bottles are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 40 mL VOA vials. These smaller vials provide enough digestate to allow one or two reanalyses when required, but reduce the amounts of samples consumed and waste generated.

VOA vials are reused if the samples they have contained have no measurable mercury above the PQL. After the previous contents of the vials have been discarded, these vials are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated vials) or below the PQL (uncontaminated vials). Labels are removed from the vials by wiping

with a paper towel saturated with toluene. Uncontaminated vials are rinsed with laboratory grade reagent water. Contaminated vials are discarded.

The Pyrex media bottles in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this spreadsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.3 Using an industrial marker with super permanent ink, label clean VOA vials with the appropriate sample numbers and standard identifications for each sample, preparation blank, laboratory control spike and matrix spike and standard to be digested.

7.4 Calibration Preparation - Use a bottle-top dispenser to add 100 mL of laboratory grade reagent water to a standard digestion bottle (250 mL media bottles). Using a calibrated adjustable pipette, prepare the high calibration standard by adding 1000 uL of Intermediate Mercury Standard A to an appropriately labeled media bottle containing 100 mL of laboratory grade reagent water. The mercury concentration of this calibration standard is 10.0 ug/L. Calibration levels 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L are made by diluting the 10.0 ug/L standard into calibration blank solution. See below for amounts. The 0.2 ug/L and 5 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

Calibration Level	Amount added	Amount calibration blank solution
0.2 ug/L	0.3 mL	14.7 mL
0.5 ug/L	0.5 mL	9.5 mL
1.0 ug/L	1 mL	9 mL
5.0 ug/L	5 mL	5 mL

7.5 Add 100 mL of laboratory grade reagent water to the media bottle labeled “ICV”. Using a calibrated adjustable pipette, prepare the Initial Calibration Verification standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B

to the water in this bottle, and record the bottle number in the Mercury Preparation Logbook. The mercury concentration of the ICV is 6.0 ug/L.

7.6 Prepare an appropriate number of preparation blanks (PBW) by adding 25 mL of laboratory grade reagent water to labeled vials.

7.7 Prepare an appropriate number of laboratory control samples (LCSW) by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25 mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L.

7.8 Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.

7.9 Preparation blanks, laboratory control spikes and matrix spikes are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the media bottles are four times those listed in sections 7.10 through 7.13 but the standards are not heated.

SAMPLE PREPARATION AND DIGESTION

7.10 Using a graduated disposable dosecup, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add 1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, swirling to mix after each addition. Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.

7.11 Add 2 mL of potassium persulfate solution to each sample. Cap the vials and place them in a preheated water bath. Monitor the temperature of the bath with a spirit thermometer throughout the digestion. The temperature of the water bath will fall below 95^{o} C upon addition of the digestion vials. After the temperature of the bath has risen back to 95^{o} C, continue heating the samples at 95^{o} C for two hours. Record initial and final digestion times and temperatures in the mercury prepareation benchsheet.

7.12 Remove bottles from the water bath and allow to cool to room temperature. If the purple permanganate color has failed to persist after digestion in any of the

samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 95^{o} C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the second heating step, consult the Department Manager for advice on how to proceed.

7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the sample will change from purple to colorless. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.14 Digested mercury samples are analyzed using the CETAC M-6100 Automated Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace Mercury Analyzer software running on a dedicated PC. Detailed instructions for setting up the instrument and analyzing samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.15 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.15.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a

separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{MC \times DF \times IV}{FV}$$

where: MC = Measured mercury concentration (ug/L)
DF = Dilution factor at instrument
IV = Initial sample volume (mL)
FV = Final digestate volume (mL)

7.17 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.

7.18 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a "U" qualifier.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the

Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of a laboratory grade reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory grade reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The intermediate standards used for preparing the calibration standards are prepared at least once per month in 2% nitric acid. Because mercury may be adsorbed onto the walls of glass and plastic containers, the calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110%

of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 80% to 120% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within

80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

8.12 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.13 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 7470 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (9/94), Method 7470A.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.2, 10/25/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies.

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

List of Tables and Figures

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TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient $\geq$ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within $\pm$ 30% of true value.	Correct problem and repeat calibration.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within $\pm$ 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within $\pm$ 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery $\pm$25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery $\pm$ 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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TABLE 2

DOD QSM VERSION REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	(Refer to current revision of SOP QA-806)				
LOQ establishment and verification	(Refer to current revision of SOP QA-806)				
Initial calibration (ICAL) for mercury - minimum 5 standards and a calibration blank	Daily ICAL prior to sample analysis.	5 points plus a calibration blank, r ≥ 0.995.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 2

DOD QSM VERSION REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	Problem must be corrected. All samples following the last acceptable calibration blank must be reanalyzed.
LCS	One per preparatory batch.	Water: Recovery must be within + 20% of the true value Soil: Recovery must be within vendor supplied limits (varies by lot).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	Recovery must be within + 20% of the true value.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: Recovery must be within + 20% of the true value. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 3

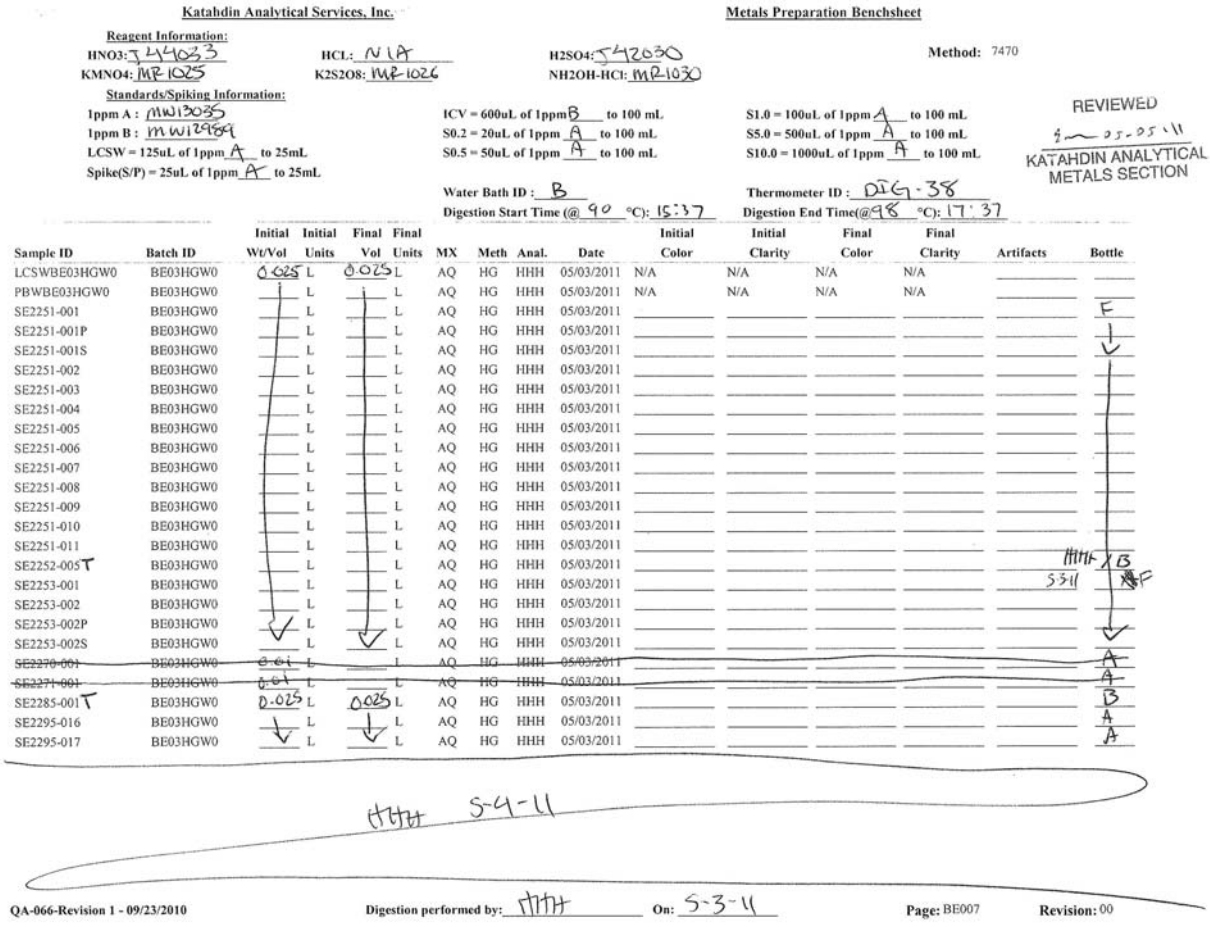
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-08	USEPA METHOD 7470
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	1)Sampling and gas stream switching performed automatically by mercury analyzer. 2)Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	1)Sampling and gas stream switching performed manually by analyst. 2)Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	1) Known reference sample (ICV) analyzed daily. 2) Calibration verified after every 10 samples with CCV.	1) Known reference sample analyzed quarterly. 2) Calibration verified after every 20 samples.
QC - Calibration Blanks	Acceptance criteria employed for 245.1: ± PQL	Acceptance criteria stated in 245.1: ± MDL

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
HNO3: J440335 HCL: N/A H2SO4: J42030
KMNO4: MP-1025 K2S2O8: MP-1026 NH2OH-HCl: MP-1030

Method: 7470

Standards/Spiking Information:
1ppm A : MW13035
1ppm B : MW12989
LCSW = 125uL of 1ppm A to 25mL
Spike(S/P) = 25uL of 1ppm A to 25mL

ICV = 600uL of 1ppm B to 100 mL
S0.2 = 20uL of 1ppm A to 100 mL
S0.5 = 50uL of 1ppm A to 100 mL

S1.0 = 100uL of 1ppm A to 100 mL
S5.0 = 500uL of 1ppm A to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL

REVIEWED 5-05-11 KATAHDIN ANALYTICAL METALS SECTION

Water Bath ID : B
Digestion Start Time (@ 90 °C): 15:37
Thermometer ID : DIG-38
Digestion End Time(@ 98 °C): 17:37

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWBE03HGW0	BE03HGW0	0.025	L	0.025	L	AQ	HG	HHH	05/03/2011	N/A	N/A	N/A	N/A		
PBWBE03HGW0	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011	N/A	N/A	N/A	N/A		
SE2251-001	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						F
SE2251-001P	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-001S	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-002	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-003	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-004	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-005	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-006	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-007	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-008	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-009	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-010	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-011	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2252-005T	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011					HHH 5-3-11	B
SE2253-001	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2253-002	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2253-002P	BE03HGW0	↓	L	↓	L	AQ	HG	HHH	05/03/2011						↓
SE2253-002S	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
~~SE2270-001~~	~~BE03HGW0~~	~~0.01~~	~~L~~		~~L~~	~~AQ~~	~~HG~~	~~HHH~~	~~05/03/2011~~						~~A~~
~~SE2271-001~~	~~BE03HGW0~~	~~0.01~~	~~L~~		~~L~~	~~AQ~~	~~HG~~	~~HHH~~	~~05/03/2011~~						~~A~~
SE2285-001T	BE03HGW0	0.025	L	0.025	L	AQ	HG	HHH	05/03/2011						B
SE2295-016	BE03HGW0	↓	L	↓	L	AQ	HG	HHH	05/03/2011						A
SE2295-017	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						A

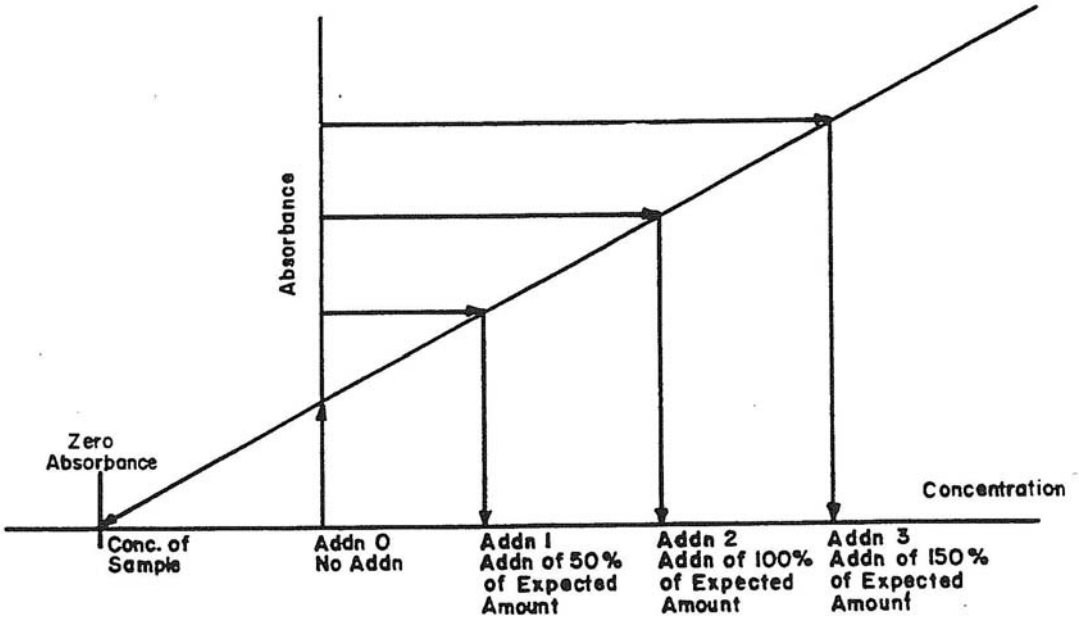
HHH 5-4-11

QA-066-Revision 1 - 09/23/2010 Digestion performed by: HHH On: 5-3-11 Page: BE007 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 2

STANDARD ADDITIONS PLOT



ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: G. Brewer

Review Date: 06/14/16

SOP Number: CA-615-02

SOP Title: Mercury Analysis of Aqueous Samples by Method 7470

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: G. Brewer **Date:** 06/14/16

QAO Signature: Susie Dimond **Date:** 06.14.16

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

SOP Number: CA-627
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

Prepared By: George Brewer Date: 03/01

Approved By:

Group Supervisor: George Brewer Date: 04/02/01

Operations Manager: Jol C Bunton Date: 3/29/01

QA Officer: Deborah J. Nadeau Date: 03.27.01

General Manager: Dennis P. Hufford Date: 04/03/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Changed acid solution conc. changed Run ID Naming convention added data reduction and reporting procedures updated standards tables (4-8) updated Table 10 in include ISIS configuration	LAD	02-16-05	02-16-05
02	Sect. 4.2 - changed tubing size Sect. 5 - changed acid conc.'s Sect. 7 - major changes to reflect current practises including reporting data in the metals database. Sect. 8 - major changes updating acceptance criteria. Updated tables 1, 3 & 8, 10 & 11	LAD	04/06	04/06
03	Updated Tables 4, 5 and 6 with current standards Updated Table 1 with serial dilution, Post Digestion Matrix spike, MSA, ICS-A, ICS-AB and IDL minimum frequency or criteria. Updated Sect. 8 regarding client specific requirements.	LAD	07/07	07/07
04	Section 7.18-changed instrument identifier to reflect new instrument; section 8-changed acceptance criteria and ICSAB analyte list; Table 1-updated acceptance criteria and corrective actions for QC. Table 3-added all analytes to list-removed "for information only" list.	LAD	04/08	04/08
05	Updates to reflect changes from 6020 to 6020A. Added Hardness by calculation attachment. Added LL QC requirement and criteria to Sect 8 and Table 1. Added criteria to analyze PQL Std. at beginning and END of run.	LAD	02/09	02/09

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 8 and QC Tables - Added DoD QSM references and criteria. Section 10 - Added references. Tables 4 → 7 - Added information pertaining to CCV conc. change	LAD	08/09	08/09
07	Added Table 2 with DoD QSM ver. 4.1 QC requirements. Updated Section 4.1, Table 10 and Table 11 with new autosampler information.	LAD	04/10	04/10
08	Sect. 1.1 - Added definitions. Sect. 4.1, 4.2, 5.2 7.9, 7.10, 7.1, 7.16 and 8.7 - minor changes to reflect current practice. Sect. 9 - added MDL, LOD and LOQ information. Sect 10 - Added, ~~edited references~~. Updated Tables 1, 5, 6, 7, 8 and 9 edited references LAD 042512	LAD	04/12	04/12
09	Sect. 7 - Added reference to autosampler software, added printing calibration and removed printing of run summary	LAD	08/13	08/13
10	Sect. 7 - Updated for changes made in the Metals database for importing and handling data. Sect. 10 - updated and added references. Added Table 3 - DoD QSM 5.0 QC Requirements	LAD	06/14	06/14
11	Sect. 7, Table 1, 2, 3, 6, 8 & 11 - Updated to reflect change from 5 pt. to 2 pt. calibration. Table 7, 8 & 9 - Updated to reflect change in Aluminum PQL	LAD	04/16	04/16

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-627-11**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**
.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-627-11**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-ppb (ug/L) concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability Method 6020 in a multi-laboratory study on solid wastes are listed as “analytes” in Table 4. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, and operating conditions. If Method 6020 is used to determine any analyte not listed in Table 4, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards.

1.1 Definitions:

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-MS - Inductively Coupled Plasma Mass Spectrometry.

ICS - Interference Check Samples - Two standards (ICS-A and ICS-AB) used to verify the effectiveness of interference correction equations. Solution ICS-A contains only interferents (Al, Ca, Fe, Mg, Na, K, P, S, Mo, Ti, C, Cl) at high

concentrations; solution ICS-AB contains interferents at the same concentrations as well as analytes at low (20 ug/L) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence.

Internal Standard - Pure analytes added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes that are components of the same sample or solution. Internal standards must be analytes that are not native to the sample.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LDR - Linear Dynamic Range - The concentration range over which the instrument response to an analyte is linear.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.
LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

Post-Digestion Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP-MS analysis by USEPA Method 6020 who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP-MS analysis by USEPA Method 6020 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Spilled samples and reagents should be cleaned up from instrument and laboratory surfaces immediately.

Liquid argon represents a potential cryogenic and suffocation hazard and safe handling procedures should be employed at all times when handling liquid argon

tanks and fittings. Safety glasses and cryogenic-resistant gloves should be worn when changing or adjusting argon tanks.

The Agilent 7500 ICP-MS spectrometer is safety-interlocked to prevent user exposure to harmful electrical voltages, radio frequency emissions, ultraviolet radiation, high temperatures, and other hazards. At no time should the operator attempt to disable these interlocks or operate the instrument if any safety interlock is suspected to be disabled

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention and waste minimization techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in ICP-MS spectrometry may contain high concentrations of acids and toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested samples and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Instrument lab. Further information regarding waste classification and disposal may be obtained by consulting Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples that require total (“acid-leachable”) values must be digested using appropriate sample preparation methods (such as USEPA Methods 3005 - 3051).

2.2 USEPA Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled argon plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of a vacuum interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 INTERFERENCES

Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). The Agilent 7500 ChemStation data system is used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences which could affect ICP-MS determinations have been identified. Examples include $ArCl^+$ ions on the As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotopic abundances from the literature, the most precise coefficients for an instrument must be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar\ ^{35}Cl^+$ signal):

Corrected ^{75}As signal (using natural isotopic abundances for coefficient approximations) = (m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal),
where the final term adjusts for any selenium contribution at 77 m/z.

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes or ^{82}Kr from krypton contamination in the Ar).
Similarly:

Corrected ^{114}Cd signal (using natural isotopic abundances for coefficient approximations) = (m/z 114 signal) - (0.027) (m/z 118 signal) - (1.63)(m/z 108 signal),
where last 2 terms adjust for any tin or MoO^+ contributions at m/z 114.

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108. Also, use of m/z 111 for Cd is even subject to direct ($^{92}ZrOH^+$) ions and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are only illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<l percent) counting precision.

The interference correction equations that are used by this laboratory in performing USEPA Method 6020 are listed in Table 5. The accuracy of these types of equations is based upon the constancy of the observed isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the “parent” ion have not been found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based

upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas have been shown to greatly reduce molecular interferences (the Agilent 7500 ICP-MS spectrometer employs spray chamber cooling to effect aerosol desolvation). These techniques can be used provided that method detection limit, accuracy, and precision requirements for analysis of the samples can be met.

3.1 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) are recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. The internal standard used should differ from the analyte of interest by no more than 50 amu. See Table 15 for a list of internal standards used. When the intensity level of an internal standard is less than 70 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

3.2 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 APPARATUS AND MATERIALS

4.1 Agilent 7500 ICP-MS system, consisting of the Agilent 7500 ICP-mass spectrometer and its controlling computer data station. The spectrometer is capable of providing resolution better than or equal to unit resolution at 10% peak height. The Agilent 7500 mass range of 2-260 amu exceeds the method requirement of 2- 240 amu. The Agilent 7500 ChemStation software allows automatic corrections for isobaric interferences and correction for internal standard responses as required by the method. All critical argon flows including nebulizer argon are under mass flow controller control and a peristaltic pump is used for sample introduction. Peripheral equipment includes a Elemental Scientific SC-4 PX

Fast Autosampler and Sample Introduction system, and Bullzip PDF printer set to print to file ICPMS_CP.pdf located in folder PDF_PRINTS on the desktop.

4.2 Peristaltic pump tubing – 2-stop ESI PVC flared black-black (0.76 mm ID) and orange-green (0.38 mm ID). 3-stop Pharmed blue-yellow (1.52 mm ID).

4.3 15 ml 17x100 mm polypropylene or polystyrene disposable test tubes for samples and 50 ml polypropylene centrifuge tubes for standards.

4.4 Automatic adjustable-volume pipetters of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Trace metal grade pipette tips.

4.6 Volumetric glassware or plasticware of suitable precision and accuracy.

4.7 Talc free vinyl gloves.

4.8 Argon gas supply (high purity grade gas or liquid, 99.99%).

4.9 For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust etc. A clean laboratory work area, designed for trace element sample handling must be used. Standards, samples and blanks should be exposed to the laboratory environment as little as possible. The use of preparation blanks and spikes should be used to verify the absence of sources of contamination and loss. If necessary, polypropylene sample tubes should be rinsed and stored in dilute acid prior to use.

NOTE: Chromic acid must not be used for cleaning glassware for trace metals analysis.

5.0 REAGENTS AND STANDARDS

5.1 Acids used in the preparation of standards and for sample processing must be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Mallincrodt/Baker “Instra-Analyzed” trace-metals grade acids are appropriate. It is important to match the acid concentration in standards and samples. Concentrations of antimony and silver between 50-500 ug/L require 1% (v/v) HCl for stability; for concentrations above 500 ug/L additional HCl will be needed. For this reason, it is recommended that antimony and silver concentrations in samples and standards be maintained below 500 ppb wherever possible. Acids are received in poly-coated glass bottles, and are stored under the hood in the Metals sample preparation laboratory at room temperature until use. All

acids are considered to have a shelf life of three years from date of receipt unless otherwise indicated by the vendor. Refer to the current revision of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details.

5.2 Laboratory reagent grade water, trace metals free, equivalent to ASTM Type 1 (ASTM D 1193), >18 Megohm/centimeter resistivity.

5.3 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 6 and 7 for a listing of stock standards required, and to Table 8 for element concentrations in stock standards. Purchased stock standards are received in polyethylene containers and are stored in their original containers at room temperature in the Metals Standards Preparation Laboratory. All purchased stock standards are given an expiration date as indicated by the manufacturer. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.4 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 7 for a listing of intermediate standards required and for preparation instructions. Refer to Table 8 for element concentrations in intermediate standards. Intermediate standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. Intermediate standards are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.5 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument, to provide internal standardization through on-line addition, and to perform all necessary QC checks. Refer to Table 6 for a listing of working standards and for preparation instructions. Refer to Table 8 for element concentrations in working standards. Working standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. All working standards except the ICSA and ICSAB solutions are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. The ICSA and ICSAB solutions are assigned an expiration date of one week from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.6 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

5.6.1 The calibration blank consists of the same concentrations of the same acid(s) used to prepare the final dilution of the analyte calibration solutions (currently 1% HNO_3 and 0.5% HCl, v/v, in laboratory reagent grade water). Use of HCl for antimony and silver is cited in Section 5.1.

5.6.2 The preparation blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the associated digested sample solutions.

5.6.3 The rinse blank consists of 4% HNO_3 and 0.5% HCl,v/v, in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP-MS should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Instrument control and data acquisition are completely automated through the use of the Agilent Chemstation software. The main Chemstation screen is accessed by double-clicking the “ICP-MS Top” icon on the Windows desktop. Autosampler tables are edited by selecting “Edit Sample Log Table” from the Sequence menu in the Agilent Chemstation software. In the following discussion, software menu items that are to be selected are printed in boldface. The instrument operating conditions, acquisition parameters, acquisition masses, and internal standards for analysis USEPA Method 6020 are detailed in Table 12.

7.2 Turn on the argon supply at the tank and set the pressure to >700 kPa.

7.3 Turn on the water chiller/recirculator.

7.4 Verify that the exhaust hood is in operation.

7.5 Ensure that the internal standard solution bottle is adequately full. Consumption is approximately 2.5 mL/hour.

7.6 Verify that the rinse station reservoir has an adequate supply of reagent water.

7.7 Verify that the drain reservoir has adequate room to accept the day's drain waste. Empty the reservoir as necessary into an appropriate waste container (Waste Stream A) located in the Hazardous Waste Storage Area.

7.8 Inspect the peristaltic pump tubes for signs of flattening and wear, and replace them as necessary. Clamp the peristaltic pump tubes into the peristaltic pump.

7.9 Open ESI autosampler software by double-clicking the "ESI SC" icon. Open the Chemstation software by double-clicking the "ICP-MS Top" icon. Initiate the plasma by selecting **Instrument>>Instrument Control>>Plasma>>Plasma On** and allow the instrument to aspirate calibration blank solution for at least 45 minutes. During this warm-up, select **Tune>>Sensitivity>>Start** to start the instrument scanning the mass range. Verify that the flow of sample and internal standard solutions through the uptake lines and into the nebulizer is free from pulsations by introducing an air bubble into each line and observing its progress. Adjust the pump clamp tension on each line to obtain a constant, pulse-free flow.

7.10 After the 45 minute warm-up, check the responses of masses 82 and 83 to insure minimal krypton intereference with selenium. Mass 83 response should be < 2000 counts per second. Then aspirate the Instrument Tune Solution (10 ppb Li, Y, Ce, Tl) and check the responses and RSDs at masses 7, 89, and 205.

7.11 Generate a tune report by selecting **Tune>>File>>Generate Report**. Evaluate the tune report against the tune specifications listed in Table 12. If the tune passes all specifications, proceed to step 7.14.

7.12 If the tune report indicates unacceptable instrument performance for any parameter, initiate an autotune by selecting **Tune>>Autotune>>Run**. The Chemstation software will attempt to tune the instrument to meet the tune specifications, and will generate a new tune report after autotuning. Evaluate the new tune report against the tune specifications listed in Table 13.

7.13 Repeat step 7.12 until all tune specifications have been met. File the final tune report.

7.14 Aspirate the P/A tuning solution (see Table6) and run a P/A auto tune by selecting **Tune>>Tune>>P/A Factor>>Run**. This will calibrate the pulse and analog modes of the detector. File the P/A report with the Tune report.

7.15 Load the instrument analytical method and calibrations table for USEPA Method 6020 into memory by selecting:

Methods>>Load>>K1PTCAL16.M>>K1PTCAL16.C.

7.16 Edit the sequence template "K6020.S" to create an analytical sequence table listing all of the samples to be analyzed. To do this, select "Edit Sample Log Table" from the **Sequence** menu in the Agilent Chemstation software. Double-click **SMPL** from the menu at the top left. Fill in the sample table with sample IDs, vial numbers, analytical method (K1PTCAL16.M for all samples), dilution factors, and failure actions. When the sample table is complete, select **Print** to print this table. Close the "Edit Sample Log Table" window. Save the sample log table under a new name by selecting **Save** under the **Sequence** menu and then typing the name.

7.17 Load the autosampler racks according to the analytical sequence printout.

7.18 Select **Sequence>>Load and Run Sequence**, and select the appropriate autosampler sequence table from the displayed list. Enter the analyst's initials in the Operator box. Change data file name to appropriate designation. The protocol for naming data files is as follows: the 1st character is a letter that identifies the instrument ("J" for the Agilent 75000 ICP-MS), the 2nd character is a letter that identifies the year of the run ("G" for 2013, "H" for 2014, etc.), the 3rd character is a letter that identifies the month of the run ("A" for January, "B" for February, etc.), the 4th and 5th characters are numbers that identify the date of the run ("01" for the first day of the month, etc.), and the 6th character is a letter that sequentially identifies the run ("A" for the first run of the day on that instrument, "B" for the second run, etc.). For example, the run identified as "JGA16A" is the first run of the day that was performed on January 16, 2013, using the Agilent 7500 ICP-MS. Select **Run**. The instrument will analyze all samples in the order listed in the table. Analysis must proceed in the sequence described in Table 11 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of three replicate scans is required for all standards and samples. Analysis always begins with the analysis of a calibration blank followed by at least three multielement calibration standards to calibrate the instrument. The system is flushed with rinse blank between each sample and standard, and each sample and standard is aspirated for at least one minute prior to the beginning of mass scanning.

7.19 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.20 A practical quantitation limit standard (PQL) is analyzed at the beginning of the run to verify calibration accuracy at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.21 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples,

and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.22 Interference check standard solutions ICS-A and ICS-AB must be analyzed at the beginning of each run and every 12 hours to verify the adequacy of interference corrections. Refer to Section 8 and Table 1 for additional information.

7.23 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a calibration verification sample (ICV, ICB, CCV, or CCB) for that element must not be reported, except as noted in Sections 8.5, 8.6, and 8.9. The sample must be reanalyzed for the element in question.

7.24 All samples that exceed the linear dynamic range must be diluted and reanalyzed.

7.25 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the sample log table prior to initiation of analysis.

7.26 If an element has more than one monitored isotope, examination of the concentration calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes. In the case of Pb, quantitation is based on the sum of isotopes 206, 207 and 208 to compensate for any variation in naturally occurring isotope ratios. This is accomplished through the use of the interference correction equation for lead.

7.27 Calculations, aqueous samples: Final element concentrations for aqueous samples are reported in units of micrograms per liter (ug/L). The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (ug/L)} = \frac{MC \times DF \times FV}{IV}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
IV = Digested sample volume (L)

7.28 Calculations, solid samples: Final element concentrations for solid samples are reported in units of milligrams per kilogram (mg/kg) on a dry weight basis. The

reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (mg/kg dry weight)} = \frac{MC \times DF \times FV \times 100}{W \times S}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
W = Weight of digested wet sample (g)
S = Percent solids

DATA REDUCTION AND REPORTING

7.29 Follow these steps to create the data import file: Open the FileView program using the "FIVIEW" icon on the Windows Desktop. Select "Data" in left window. Select the data file of interest and double click to move the required samples into the "Process List". Make sure the "Corrected Data" box is checked. Click the "Process" button. The data will be displayed in a spreadsheet format.

7.30 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "K2008.sbl" from this list of options and click "open." Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the analyte masses in the spreadsheet.

7.31 Select "Quant Info" from the top menu and select "Quant Results" from the displayed options. This will display the data in concentration units.

7.32 Select the "Transpose" from the menu. Click on "file" within the chart to highlight the data.

7.33 Select "Tools" from the top menu and "Copy Selected Data to CSV File" from this list of options. Set the name to the file as "FileName.CSV", e.g., "JGA28A.CSV". Save the file to the ICP-MS DATA folder on metals on server_a.

7.34 Rename the pdf file to the appropriate file name in the PDF_Prints window and save to J-ICMS-Data file in My Network Places. Right click on ICPMS_CP.pdf icon to copy and past blank file into PDF_Prints window for the next run.

To import data into the Metals Database:

7.35 Open the data file from metals on Server_a. Replace dashes in Cal Blank line with zeros. Replace dashes in Cal Std 1 line with ~~0.5~~ 50 for most all elements. Change aluminum and silicon with ~~10~~ 1000 and change sodium, magnesium, potassium, calcium, and iron with ~~100~~ 10,000. All cells under metals with ###, replace with 999999. Save file in ICP-MS Data folder on Metals on Server a. Select the "ICPMS

Import" icon from the Windows Desktop, the ICPMS Import window will appear. Enter the datafile name without extension, (e.g., "JGA28A") and click ~~the "Import" button~~ "ok."

7.36 When the "Import finished" message appears, close the ICPMS Import window and select the "KIMS_METALS" icon from the Windows Desktop. The Metals Database Main Menu will appear. Select Additional Data Handling and then select Accept Samples by Element. Type in file name and reject any items that fail run QC.

7.37 Select the "Reporting Menu" button. From the Reporting Menu screen select the Batch QC Menu button and then the "Calculate Batch QC" button.

7.38 From the resulting list of QC results, deselect any items that fail run QC. Click on the "Accept Selected Batch QC" button.

7.39 From the Metals Main Menu, select the "Additional Data Handling" button. The Data Menu will appear. Select the "Report Added Compounds" button.

7.40 From the resulting list of sample results, deselect any items that fail run QC. Click on the "Accept Data" button.

7.41 Once all associated data from an analysis run have been processed, go to the RUNLOG INFO table of the metals database. Sort for the file of interest. Add lines for the 6020 and 200.8 Method Tunes. Change the time column accordingly. Go to the Generate Coverage portion of the Export Menu and print the Run Log and Logbook Page for the analysis run.

7.42 To extract Tune Reports and P/A Factor Tuning Report click on metpdf on Imageserver icon. Select J-ICPMS Data folder and select file on interest. Select Document drop down menu>pages>extract>select page numbers and click ok. Close document and save in metdpdf on "imageserver (P:) in J-ICP-MS-INST Tune folder as Filename+Tune.

7.43 Remove "blanks" and "rinses" from pdf file by selecting Document drop down menu>pages>delete>select appropriate pages at the beginning and end of report. Save document with "RAW" added to the end of the file name. Save in the "ICPMS DATA" section of the "METPDF" directory on the IMAGESERVER.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6020 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does

not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent blank, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.9) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

ANALYTICAL RUN QC SAMPLES

8.4 Initial instrument calibration: The instrument is calibrated by running a calibration blank and at least three multielement calibration standards. For each element, calibration is performed fitting a single order equation to the calibration data, as follows:

$$Y=aX + [Blank]$$

where: Y = Concentration (ug/L)
X = Measured signal intensity (counts per second)
a = Slope of the calibration line
[Blank] = Measured signal intensity of the calibration blank

Fitting the calibration equation through the measured intensity of the calibration blank, rather than through the y-intercept of the line, provides the best calibration accuracy at the low end of the calibration range. When this equation is used, however, the Agilent software does not calculate a calibration coefficient. For this reason, calibration accuracy at the high end of the calibration range is checked by reanalyzing the highest calibration standard as a sample immediately after instrument calibration. Recoveries for all elements must be within 90% to 110% of the true value in the high calibration standard. If the high calibration standard fails, result for the failing elements may not be reported from the run until the problem is corrected and a passing high calibration standard has been analyzed. Calibration accuracy in the middle of the calibration range is verified by analysis of the CCV solution (see Section 8.6 below). Calibration accuracy at the reporting limit is verified by analysis of the PQL Check Standard (see Section 8.7 below).

8.5 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 70 and 120 percent of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. The sample must be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal standard intensities fall within the prescribed window. The intensity levels of the internal standards for the calibration blanks (ICB and CCBs) and calibration verification standards (ICV and CCVs) must agree within ± 20 percent of the intensity level of the internal standard of the original calibration solution. If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.6 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from standard sources different than those of the calibration standards and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the

ICV fails, result for the failing elements may not be reported from the run, unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

8.7 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements in samples bracketed by the failing CCV may not be reported, unless the CCV recovery is greater that 110% and the sample result is less that the PQL. For DoD analyses, results may not be reported without a valid CCV or report flagged results if reanalysis is not possible.

8.8 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning of each run (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are one-fifth the laboratory's practical quantitation limit (assuming a 5-fold dilution of all digestates during analysis). Element recoveries for the PQL Check Standard must fall within 70% to 130% of the expected values (unless the samples analyzed are for the Department of Defense (80% to 120% recovery limits) or other client-specific limits are imposed). If the PQL Check Standard fails, results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than the high limit and the sample result is less than the PQL.

8.9 A calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the applicable reporting limit (or PQL) for each element. The reporting limit should be determined on a project specific basis, taking into account the data quality objectives for the project. This information must be communicated through a project QAPP and through the Katahdin project manager. When no project specific reporting limit is specified, the laboratory PQL shall be used. Some project specific limits may require reporting down to the MDL or IDL and taking corrective action based on these levels. Results that fall between the PQL and the IDL or MDL must always be flagged as "estimated" with a "J".

8.10 If an ICB or a CCB fails the acceptance criteria of less than the reporting limit, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for an ICB or CCB is greater than the PQL (or reporting limit), sample results that are less than the PQL (or reporting limit) or that are greater than or equal to ten times the measured ICB or CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

8.11 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantitation and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met. Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Interference check solutions ICS-A and ICS-AB are analyzed at the beginning of each run and at least every 12 hours during the run to verify the effectiveness of interference corrections. Solution ICS-A contains high concentrations of interferents (Al, Ca, Fe, Mg, Na, P, K, S, C, Cl, Mo, and Ti) only. These should recover between 80% and 120% of the true value. The measured concentrations of other elements in this solution should be very low, indicating that interfering mass correction equations are adequate. Solution ICS-AB contains interferents at the same high concentrations, and all other analytes at 20 ug/L. Measured recoveries for all analytes should be within 80% to 120% of the true values.

PREPARATION BATCH QC SAMPLES

8.12 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spiked sample, or matrix spiked sample duplicate.

8.12.1 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) (or project specific reporting limit, if applicable) for each element. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL or reporting limit, associated sample results that are less than the PQL or reporting limit or that are greater than or equal to ten times the measured preparation blank concentration may be reported.

8.12.2 A laboratory control sample (LCSW, LCSO, or LCSS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for

solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the recovery of a laboratory control sample is greater than 120%, associated sample results that are less than the PQL or reporting limit may be reported.

SAMPLE MATRIX QC SAMPLES

8.13 The relative percent difference (RPD) between matrix duplicate, matrix spike duplicate, or laboratory control duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = First sample or LCS result
D_2= Second (duplicate) sample or LCS result

A control limit of 20% RPD is applied to duplicate analysis, if the result is greater than 100 times the instrument detection limit. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.14 The recovery for each element in a spiked sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If a recovery fails, the associated sample result must be flagged on the report of analysis. The spike recovery should be calculated as follows:

$$Recovery\ (\%) = \frac{S-U}{SA} *100\%$$

where: S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

8.16 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), the measured concentration of a five-fold dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} *100\%$$

where: L = Serial dilution result (corrected for dilution)

S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The action taken is dependent upon project requirements. The associated sample result may be flagged on the report of analysis, the sample may be reanalyzed at dilution to eliminate the interference, or a post-digestion spike may be performed (see section 8.16).

8.17 An analyte spike that is added to an aliquot of a prepared sample, or its dilution, should be recovered within 80% to 120% of the known value if the result for the unspiked aliquot is less than four times the amount of spike added. If the post-digestion spike is not recovered within these limits, the sample should be diluted and reanalyzed to compensate for the matrix interference or the method standard additions may be employed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of USEPA Method 6020 for other method performance parameters and requirements.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6020A.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.2, 10/25/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

Agilent 7500 ICP-MS ChemStation Operator's Manual, Agilent Technologies, Inc., 2000.

List of Tables

TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	If more than 1 caibration std is used, correlation coefficient (r) $\geq$ 0.998	Recalibrate
Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	Do not use results for failing elements, unless ICV >110% and sample result < PQL/reporting limit.
Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL or project specific reporting limit.	Do not use results if sample $\geq$ PQL/reporting limit and < 10x ICB level.
PQL Standard or LLCCV	At beginning and end of run	70-130% of true value	Do not use results for failing elements, unless PQL rec.> upper limit and sample result < PQL/reporting limit.
Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within $\pm$ 10% of true value.	1) Do not use bracketed sample results for failing elements, unless CCV >110% and sample result < PQL/reporting limit. 2) Investigate and correct problem.
Continuing Calibration Blank (CCB)	Immediately after every CCV	Absolute value of CCB < PQL or project specific reporting limit.	Do not use sample results if $\geq$ PQL/reporting limit and < 10x CCB level.
Interference Check Solution A (ICS-A)	Before analyzing samples, and every 12 hours during a run.	Interferents: Recovery within $\pm$ 20% of true value. Analytes: No criteria established (Project specific criteria may apply)	Do not use sample results for failing elements.
Interference Check Solution AB (ICS-AB)	Before analyzing samples, and every 12 hours during a run.	Recovery within $\pm$ 20% of true value.	Do not use sample results for failing elements, unless ICSAB >120% and sample result < PQL/reporting limit.
Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL (standard practice), or based on the project specific guidelines.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and <10x the blank conc.
Laboratory Control Sample (LCSW/LCSS/LCSO)	At least one per digestion batch of 20 or fewer samples.	Recovery within $\pm$ 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples, unless LCS >120% and sample result < PQL.

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TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Duplicate Sample (D), Matrix Spike Duplicate (P), or LCS Duplicate (LC2W/LC2S/LC2O)	See section 8.11	1) RPD ≤ 20%, if sample > 100x IDL.	Flag results
Post-Digestion Matrix Spike (A)	When serial dilution fails and analyte concentration < 100 x MDL.	Recovery ± 20% of true value, if sample < 4x spike added.	Flag results and/or analyze sample by method of standard additions.
Serial Dilution (L)	1 per digestion batch	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.
Internal Standard (IS)	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte.	1) For each sample, IS intensity within 70%-120% of that of initial calib. blank. 2) For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calib. blank.	Do not use results for failing elements.
Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL at least 2-3x IDL	1) Repeat IDL study. 2) Raise PQL.
Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Reevaluate PQLs
Method of Standard Additions	When matrix interference is suspected	r ≥ 0.995	Dilute and reanalyze sample to eliminate interference.

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TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Instrument detection limit (IDL) study	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Tuning	Prior to ICAL.	Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height; For stability, RSD ≤ 5% for at least four replicate analyses.	Retune instrument then reanalyze tuning solutions.	Flagging criteria are not appropriate.	No analysis shall be performed without a valid MS tune.
Initial calibration (ICAL) for all analytes (minimum one high standard and a calibration blank)	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r ≥ 0.995.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analytes within ± 10% of true value.	Verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All analytes within ± 10% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Low-level calibration check standard	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Linear dynamic range or high-level check standard	Every 6 months.	Within ±10% of true value.	NA.	NA.	
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For negative blanks, absolute value < LOD. Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS-A and ICS-AB)	At the beginning of an analytical run and every 12 hours.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value. May use < LOD for some projects.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes to be reported	One per preparatory batch.	Water: Recovery must be within + 20% of the true value Soil: Recovery must be within vendor supplied limits (varies by lot).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix	For matrix evaluation, use recovery must be within + 20% of the true value.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria,	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
			additional quality control tests (dilution test and post-digestion spike addition) are required to evaluate matrix effects.		determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation use recovery must be within + 20% of the true value. MSD or sample duplicate: RPD < 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution test	One per preparatory batch.	For samples with concentrations > 50 x LOQ then five-fold dilution must agree within ± 10% of the original measurement.	Perform post-digestion spike addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post digestion spike addition	When dilution test fails or analyte concentration for all samples < 50 x LOD.	Recovery within 75-125%	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Internal standards (IS)	Every sample.	IS intensity within 30-120% of intensity of the IS in the ICAL.	Flagging criteria are not appropriate.	Reanalyze sample at 5-fold dilution with addition of appropriate amounts of internal standards.	
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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TABLE 3

DoD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear Dynamic Range (LDR) or High-level Check Standard	Daily.	Within ±10% of true value.	Dilute samples within the calibration range, or re-establish/verify the LDR.	Flagging is not appropriate.	Data cannot be reported above the calibration range without an established/passing high-level check standard.
Tuning	Prior to ICAL.	Mass calibration = 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial Calibration (ICAL) for All Analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 = 0.99.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICALhas passed.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes, within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level Calibration Check Standard (Low Level ICV)	Daily.	All reported analytes within ± 20% of the true value.	Correct problem and repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the LOQ.
Internal Standards (IS)	Every field sample, standard and QC sample.	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria is met. For failed QC samples,	Flagging is not appropriate.	Samples suffering from matrix effect should be diluted until criteria are met, or an alternate IS should be selected.

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TABLE 3

DoD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
			correct problem, and rerun all associated failed field samples.		
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed. Flagging is not appropriate.	Results may not be reported without a valid calibration blank.	For CCB, failures due to carryover may not require an ICAL.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS.	All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference.

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TABLE 3

DoD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
		limits are not specified. MSD or MD: RPD of all analytes = 20% (between MS and MSD or sample and MD).			
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Only applicable for samples with concentrations > 50 X LOQ (prior to dilution). Use along with MS/MSD or PDS data to confirm matrix effects.
Post Digestion Spike (PDS) Addition	One per preparatory batch if MS or MSD fails (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%	No specific CA, unless required by the project.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Criteria apply for samples with concentrations < 50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution or post digestion spike fails and if the required by project.	NA.	NA.	NA.	Document use of MSA in the case narrative.

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TABLE 4

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-627-11	METHOD 6020, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6020: ± PQL	Acceptance criteria stated in 6020: <10% of PQL

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TABLE 5

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Element Class	Element	Sym-bol	Isotopes Monitored	Correction Equations [See note 1]
Analytes	Aluminum	Al	27	
	Antimony	Sb	121, 123	
	Arsenic	As	75	$^{75}As = (75)*1 - (77)*2.95 + (82)*2.548 – (83)*2.571$ [See note 2]
	Barium	Ba	135, 137	
	Beryllium	Be	9	
	Boron	B	11	
	Cadmium	Cd	106, 108, 111, 114	$^{111}Cd = (111)*1 – (108)*1.073 + (106)*0.764$ [See note 3] $^{114}Cd = (114)*1 – (118)*0.0268$ [See note 4]
	Calcium	Ca	44	$^{44}Ca = (44)*1 – (88)*0.0169$ [See note 7]
	Chromium	Cr	52, 53	
	Cobalt	Co	59	
	Copper	Cu	63, 65	
	Iron	Fe	54, 56, 57	$^{54}Fe = (54)*1 – (52)*0.0282$ [See note 8] $^{57}Fe = (57)*1 – (43)*0.03$ [See note 9]
	Lead	Pb	206, 207, 208	$^{208}Pb = (208)*1 + (206)*1 + (207)*1$ [See note 5]
	Magnesium	Mg	25	
	Manganese	Mn	55	
	Molybdenum	Mo	98	$^{98}Mo = (98)*1 – (99)*0.146$ [See note 10]
	Nickel	Ni	60, 61	
	Potassium	K	39	
	Selenium	Se	82	$^{82}Se = (82)*1 – (83)*1.009$ [See note 11]
	Silver	Ag	107, 109	
	Sodium	Na	23	
	Strontium	Sr	88	
	Thallium	Tl	203, 205	
	Thorium	Th	232	
	Tin	Sn	118, 120	
	Tungsten	W	182	
	Uranium	U	238	
	Vanadium	V	51	$^{51}V = (51)*1 – (53)*2.95 + (52)*0.333$ [See note 12]
	Zinc	Zn	66, 67, 68	
Internal Stan-dards.	Bismuth	Bi	209	
	Germanium	Ge	72	
	Indium	In	115	$^{115}In = (115)*1 – (118)*0.016$ [See note 6]
	Lithium	Li	6	
	Scandium	Sc	45	
	Terbium	Tb	159	
	Yttrium	Y	89	

TABLE 5 (continued)

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Notes:

1) Numbers in parentheses, e.g "(51)", indicate measured counts at the indicated mass.
2) Corrects for ArCl interference, taking into account secondary interferences from Se and Kr
3) Corrects for MoO interference, taking into account secondary interference from ^{108}Cd
4) Corrects for Sn interference
5) Corrects for variations in isotopic composition of lead
6) Corrects for Sn interference
7) Corrects for interference from $^{88}Sr^{2+}$
8) Corrects for Cr interference
9) Corrects for Ca interference
10) Corrects for Ru interference
11) Corrects for Kr interference
12) Corrects for ClO, taking into account secondary interference from Cr

TABLE 6

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Continuing Calibration Verification CCV (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.25
	ICP-MS-MIX-Z	Lab Prepared	0.50
	ICP-MS CAL 1	Lab Prepared	1.25
Calibration Standard (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.50
	ICP-MS-MIX-Z	Lab Prepared	1.0
	ICP-MS CAL 1	Lab Prepared	2.5
Initial Calibration Verification (ICV) (1.0% HNO_3 / 0.5% HCl)	CL-ICS-1,CL-ICS-4, CL-ICS-5	Spex Industries	0.20 of each
	CL-ICS-3	Spex Industries	2.0
	1000 mg/L Si	Inorganic Ventures	0.040
	1000 mg/L Al	Inorganic Ventures	0.038
	1000 mg/L B, W Solution (0.5mL each per 50mL and use same day only)	Inorganic Ventures	0.200
Practical Quantitation Limit Solution (PQL) (1.0% HNO_3 / 0.5% HCl)	ICP-MS PQL Intermediate	Lab Prepared	0.1
Interference Check Solution A (ICS-A) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
Interference Check Solution AB (ICS-AB) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
	ICP-MS-CAL 1	Lab Prepared	1.0
	ICP-MS ICSAB Intermediate	Lab Prepared	1.0
P/A Tuning Solution (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Co, Cr, Mo, Mn, Pb, Sb, Sr, U, V	High Purity Standards	0.02
	10,000 mg/L Al, K, Na	High Purity Standards	0.002
Instrument Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS-TS-2	High Purity Standards	0.10
	Conc. HNO_2	Baker Instra Analyzed	4
Internal Standard Solution (5.0% HNO_3 / 0.5% HCl)	Internal Standard Mix	Spex Industries	10

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TABLE 6

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Method Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS Method Tune Intermediate	Lab Prepared	1.0
	Internal Standard Mix 1	Spex Industries	1.0

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TABLE 7

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS PQL Intermediate (5% HNO_2/5%HCL)	10,000 mg/L K, Na	High Purity Standards or Inorganic Ventures	2.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	1000 mg/L B	High Purity Standards or Inorganic Ventures	0.40
	10,000 mg/L Al, Ca, Fe, Mg 1000 mg/L Zn	High Purity Standards	0.20 of each
	1000 mg/L As, Se, V, W, Sr, Sn, Mo, Cr	High Purity Standards or Inorganic Ventures	0.10 of each
	1000 mg/L Cu	High Purity Standards	0.06
	1000 mg/L Ba, Mn, Ni	High Purity Standards	0.04 of each
	1000 mg/L U, Be, Cd, Co, Ag, Th, Tl, Pb, Sb	High Purity Standards	0.02 of each
ICP-MS CAL 1 -(5% HNO_2/5%HCL)	1000 mg/L Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Th, Tl, U, V, Zn	High Purity Standards	0.2 of each
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.02
	Conc. HCL	Baker Instra Analyzed	2
ICP-MS-MIX-Z (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L K, Na, Fe, Mg, Ca	High Purity Standards or Inorganic Ventures	5.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.95
	1000 mg/L B, Sn, Sr, W	High Purity Standards or Inorganic Ventures	0.50 of each
ICP-MS-MIX-Y (1.0% HNO3/ 0.5% HCl)	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.030
	1000 mg/L As, Ba, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, V, Zn	High Purity Standards or Inorganic Ventures	0.30 of each
ICP-MS ICSAB Intermediate (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L Si	High Purity	0.50
	1,000 mg/L B, Sn, Sr, W	High Purity or Inorganic Ventures	0.20 each

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS Method Tune Intermediate (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Be, Co, Tl 10,000 mg/L Mg	High Purity Standards or Inorganic Ventures	0.1 of each
	1000mg/L Pb		0.30

TABLE 8

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	CCV	Cal. Std.	ICV	PQL	P/A Tune Soln.
Aluminum	500.0	1000.0	400.0	20.0	200
Antimony	25.0	50.0	20.0	0.2	200
Arsenic	25.0	50.0	20.0	1.0	
Barium	25.0	50.0	20.0	0.4	
Beryllium	25.0	50.0	20.0	0.2	
Boron	25.0	50.0	20.0	4.0	
Cadmium	25.0	50.0	20.0	0.2	
Calcium	5000.0	10000.0	4000.0	20.0	
Chromium	25.0	50.0	20.0	1.0	200
Cobalt	25.0	50.0	20.0	0.2	200
Copper	25.0	50.0	20.0	0.6	
Iron	5000.0	10000.0	4000.0	20.0	
Lead	25.0	50.0	20.0	0.2	200
Magnesium	5000.0	10000.0	4000.0	20.0	
Manganese	25.0	50.0	20.0	0.4	200
Molybdenum	25.0	50.0	40.0	1.0	200
Nickel	25.0	50.0	20.0	0.4	
Potassium	5000.0	10000.0	4000.0	200.0	200
Selenium	25.0	50.0	20.0	1.0	
Silicon	500.0	1000.0	400.0	100.0	
Silver	25.0	50.0	20.0	0.2	
Sodium	5000.0	10000.0	4000.0	200.0	200
Strontium	25.0	50.0	20.0	1.0	200
Thallium	25.0	50.0	20.0	0.2	
Tin	25.0	50.0	20.0	1.0	
Tungsten	25.0	50.0	20.0	1.0	
Uranium	25.0	50.0	20.0	0.2	200
Vanadium	25.0	50.0	20.0	1.0	200
Zinc	25.0	50.0	20.0	2.0	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 8 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Aluminum	100000	100000			
Antimony		20			
Arsenic		20			
Barium		20		10	
Beryllium		20			
Boron		20			
Cadmium		20			
Calcium	100000	100000			
Chromium		20			
Cobalt		20		10	
Copper		20			
Iron	100000	100000			
Lead		20		10	
Magnesium	100000	100000		100	
Manganese		20			
Molybdenum	2000	2000			
Nickel		20			
Potassium	100000	100000			
Selenium		20			
Silver		20			
Sodium	100000	100000			
Strontium		20			
Thallium		20		10	10.0
Tin		20			
Tungsten		20			
Uranium		20			
Vanadium		20			
Zinc		20			
Bismuth			1000.0	10	
Germanium			1000.0	10	
Indium				10	
Lithium (^{6}Li)			1000.0	10	
Scandium			1000.0	10	
Terbium			1000.0	10	
Yttrium			1000.0	10	10.0

TABLE 8 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Cerium					10.0
Lithium					10.0

1) Solution also contains 1000 mg/L Chloride, 200 mg/L Carbon, and 100 mg/L Phosphorus and Sulfur, and 2mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 9

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

ELEMENT	CONCENTRATION IN SOLUTION, mg/L					
	MS-MIX-Z	ICP-MS PQL Intermediate	ICP-MS-MIX-Y	ICP-MS Method Tune Intermediate	ICP-MS CAL 1	ICP-MS ICSAB Intermediate
Aluminum	95.0	2.0	3.0		0.2	
Antimony		0.02	3.0		0.2	
Arsenic		0.10	3.0		0.2	
Barium		0.04	3.0		0.2	
Beryllium		0.02		1.0	0.2	
Boron	5.0	4.0				0.2
Cadmium		0.02			0.2	
Calcium	500	2.0				
Chromium		0.10	3.0		0.2	
Cobalt		0.02		1.0	0.2	
Copper		0.06	3.0		0.2	
Iron	500	2.0				
Lead		0.02	3.0	3.0	0.2	
Magnesium	500	2.0		10.0		
Manganese		0.04	3.0		0.2	
Molybdenum		0.10	3.0		0.2	
Nickel		0.04	3.0		0.2	
Potassium	500	20.0				
Selenium		0.10	3.0		0.2	
Silicon	100	10.0				5.0
Silver		0.02			0.2	
Sodium	500	20.0				
Strontium	5.0	0.10				0.2
Thallium		0.02		1.0	0.2	
Tin	5.0	0.10				0.2
Thorium		0.02			0.2	
Tungsten	5.0	0.10				0.2
Uranium		0.02			0.2	
Vanadium		0.10	3.0		0.2	
Zinc		0.20	3.0		0.2	

TABLE 10

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	Instrument Calibration Standard 3 (Spex)	CL-ICS-1 (Spex)	CL-ICS-3 (Spex)	CL-ICS-4 (Spex)	CL-ICS-5 (Spex)
Aluminum		10.0			
Antimony		10.0			
Arsenic		10.0			
Barium		10.0			
Beryllium		10.0			
Boron					
Cadmium		10.0			
Calcium	1000		200.0		
Chromium		10.0			
Cobalt		10.0			
Copper		10.0			
Iron	1000		200.0		
Lead		10.0			
Magnesium	1000		200.0		
Manganese		10.0			
Molybdenum				10.0	10.0
Nickel		10.0			
Potassium	1000		200.0		
Selenium		10.0			
Silver		10.0			
Sodium	1000		200.0		
Strontium					10.0
Thallium		10.0			
Thorium				10.0	
Tin					10.0
Tungsten					
Uranium				10.0	
Vanadium		10.0			
Zinc		10.0			

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 10 (continued)

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L		
	6020ICS-0A[1] (Inorganic Ventures)	**Internal Standard Mix 1 (Spex)**	**ICP-MS-TS-2 (High Purity)**
Aluminum	1000		
Arsenic			
Cadmium			
Calcium	1000		
Chromium			
Cobalt			
Copper			
Iron	1000		
Magnesium	1000		
Manganese			
Molybdenum	20.0		
Nickel			
Potassium	1000		
Silver			
Sodium	1000		
Zinc			
Bismuth		1000	
Cerium			10000
Germanium		1000	
Indium		1000	
Lithium			10000
Lithium (^{6}Li)		1000	
Scandium		1000	
Terbium		1000	
Thallium			10000
Yttrium		1000	10000

1) Solution also contains 10000 mg/L Chloride, 2000 mg/L Carbon, and 1000 mg/L Phosphorus and Sulfur, and 20 mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 11

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Method Tuning Solution	Verify mass calibration and resolution
2	S0 (Calibration Blank)	Initial calibration
3	S1 (Calibration Standard)	Initial calibration
7	ICV (Initial Calibration Verification)	Check calibration accuracy
8	ICB (Initial Calibration Blank)	Check calibration accuracy
9	PQL (Practical Quantitation Limit)	Check calibration accuracy at low concentration
10	ICS-A (Interference Check Solution A)	Verify accuracy of mass correction equations
11	ICS-AB (Interference Check Solution AB)	Verify accuracy of mass correction equations
12	CCV (Continuing Calibration Verification)	Check calibration stability
13	CCB (Continuing Calibration Blank)	Check calibration stability
14-23	Analyze up to 10 samples	
24	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	
…	After last analytical sample, analyze PQL , followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 12

INSTRUMENT OPERATING CONDITIONS

Data Acquisition Program	Acquisition Mode	Spectrum
	Points per Mass	3
	Number of Replicates	3
	Detector Mode	Auto for all elements
	Integration Time per Point (for listed masses and their correction masses)	0.10 sec for Li, B, ^{29}Si, Sc, V, Cr, Mn, Ni, Cu, Zn, Y, Mo, Ag, In, Sn, Sb, Ba, Tb, W, Tl, Pb, Bi, Th, U
		0.30 sec for Be, As, Cd, Ge
		0.010 sec for Na, Al, K, ^{28}Si
		0.030 for Ca, Fe, Sr
		1.00 sec for Se
		0.050 sec for Mg, Co
	Spray Chamber Temperature	2^{o} C
	Total Acquisition Time	105 sec for 3 replicates
Peristaltic Pump Program	Analysis Speed	0.15 rps
Before Acquisition	Uptake Speed	0.15 rps
	Uptake Time	5 sec
	Stabilization Time	15 sec
After Acquisition (Probe Rinse)	Rinse Speed	0.15 rps
	Rinse Time (sample)	5 sec
	Rinse Time (standard)	5 sec
After Acquisition (Rinse)	Rinse Vial	1
	Uptake Speed	0
	Uptake Time	0 sec
	Stabilization Time	0 sec
Calibration Curve fit	All quantitation masses	Y=ax+(blank)
	All internal standard masses	(Excluded)
	All interference correction masses	(Excluded)
Reporting Parameters	QC Reports	On-Printer
	All Other Reports	Off

TABLE 13

INSTRUMENT TUNE SPECIFICATIONS

Sensitivity	Li >5000 cts/0.1 sec/10 ppb
	Y >10,000 cts/0.1 sec/10 ppb
	Tl >5000 cts/0.1 sec/10 ppb
Precision	Li <8% RSD (0.1 sec integration time)
	Y <5% RSD (0.1 sec integration time)
	Tl <5% RSD (0.1 sec integration time)
Oxides	<1.0%
Doubly Charged (Ce^{++}/Ce^{+})	<2.0%
Background	Li <15 cps
	Y <15 cps
	Tl <15 cps
Mass Resolution	Width at 10% peak height: 0.7-0.8 amu
Mass Axis	Li ±0.1 amu of nominal mass
	Y ±0.1 amu of nominal mass
	Tl ±0.1 amu of nominal mass

TABLE 14

METHOD TUNE SPECIFICATIONS

Precision	≤5% RSD of 4 replicates
Mass Resolution	Width at 10% peak height: <0.9 amu
Mass Calibration	±0.1 amu of nominal mass

TABLE 15

REPORTED ISOTOPES AND INTERNAL STANDARDS

ELEMENT	MASS	INTERNAL STANDARD (mass)
Aluminum	27	Scandium (45)
Antimony	123	Terbium (159)
Arsenic	75	Yttrium (89)
Barium	135	Terbium (159)
Beryllium	9	Lithium (6)
Boron	11	Lithium (6)
Cadmium	114	Yttrium (89)
Calcium	44	Scandium (45)
Chromium	52	Yttrium (89)
Cobalt	59	Yttrium (89)
Copper	65	Yttrium (89)
Iron	57	Yttrium (89)
Lead	208	Bismuth (209)
Magnesium	25	Scandium (45)
Manganese	55	Yttrium (89)
Molybdenum	98	Yttrium (89)
Nickel	60	Yttrium (89)
Potassium	39	Scandium (45)
Selenium	82	Yttrium (89)
Silicon	29	Scandium (45)
Silver	107	Yttrium (89)
Sodium	23	Scandium (45)
Strontium	88	Yttrium (89)
Thallium	203	Bismuth (209)
Thorium	232	Bismuth (209)
Tin	118	Terbium (159)
Tungsten	182	Terbium (159)
Uranium	238	Bismuth (209)
Vanadium	51	Yttrium (89)
Zinc	66	Yttrium (89)

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in “Standard Methods for the Examination if Water and Wastewater,” Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY

Prepared By: Wet Chemistry Date: 5/98

Approved By:

Group Supervisor: Faith Tanguay Date: 02/501

Operations Manager: Jol C. Butler Date: 2/01

QA Officer: Deborah J. Nadeau Date: 2·15·01

General Manager: Duncan T. Kinsman Date: 2/15/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, database and standard method reference. Other minor changes throughout.	DN	2/5·01	2/5·01
02	Added definitions. Changed concentrations for calibration curve. New instructions for instrument data system operation. New instructions for exporting data to Kims. Figures 1, 2, 5, 6, 7 updated	MRC	04.29.04	04.29.04
03	Updated acceptance criteria-Table 1. Added interferences- sect. 3. Removed references to Supervisor and ASTM II water. Added references to Department manager and laboratory reagent grade water. Added program specific information to sections. Updated figures	LAD	04/06	04/06
04	Updated Standard prep 5.4, 5.6, 5.7. Added Section 5.9 and 5.10. Updated Std. Table on pg. 7. Updated all LCS and MS recoveries to 90-110%. Changed MDL shall be performed every six months NOT annually. Updated Figure 4. Minor formatting changes throughout.	LAD	03/07	03/07
05	Added LFM recovery criteria of 90→110%. Added LCR information. Added requirement of analyzing a calibration standard at the PQL when a lower PQL is required.	LAD	08/08	08/08

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SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Deleted logbook figure & added run information figure. Added EHSM, subsampling, QA-SOP, DoD, NELAC & CA-101 references.	DM	08/09	08/09
07	Section 4 - added KI strips. Corrected prep in Sect.s 8.7 and 8.10. Minor changes to Sect. 7. Added non-conformance reports and DUP frequency to sect. 8. Removed obsolete method reference in sect. 10.	LAD	06/10	06/10
08	Sect. 7 - Added instrument shut-down instructions. Sect. 9 - Added MDL, LOD & LOQ information. Sect 10 - Added and edited references. Updated figures 1, 2, 5A, 5B, 6 & 7.	LAD	05/12	05/12

TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-728-08**, titled **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-728-08**, titled **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY.**

Recipient: _____ Date: _____

TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

1.0 SCOPE AND APPLICATION

This method details the procedure used by Katahdin Analytical Services, Inc. technical personnel for the colorimetric analysis of Nitrate/Nitrite in aqueous samples using the LACHAT automated analyzer. This SOP is applicable to samples analyzed by EPA method 353.2, Standard Methods 4500NO_3 F and LACHAT method 10-107-04-1-C. The range of the test is 0.05 to 2.0 mg/L. Samples with concentrations higher than this range require dilution prior to analysis.

1.1 Definitions

Method Blank - A laboratory reagent grade water sample that is carried through the entire analytical procedure in the same manner as a sample.

LCS/ICV - Laboratory Control Sample/ Initial Calibration Verification. One LCS/ICV per batch is prepared from a separate source from the CCV and calibration curve standards. LCS/ICV verifies the calibration curve.

CCV - Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is run every ten samples.

CCB - Continuing Calibration Blank. The CCB is laboratory reagent grade water with no reagents added. One CCB is run every ten samples.

Linear Calibration Range (LCR) – The concentration range over which the instrument response is linear.

Matrix Spike (MS) or Laboratory Fortified Sample Matrix (LFM) – A known amount of analyte is added to 10% of routine samples. The added analyte concentration should be the same as that used in the LCS.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis Nitrate/Nitrite by LACHAT Auto Analyzer. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of Nitrate/Nitrite by LACHAT Auto Analyzer to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets (MSDSs) is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health & Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health & Safety Manual and SOPs SD-903, “Sample Disposal” and CA-107, “The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and Standards,” current revisions. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

2.0 SUMMARY OF METHOD

Nitrite is determined on channel 2 of the LACHAT Auto Analyzer by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. This results in a water soluble magenta color which is read at 520 nm. Nitrate is reduced to Nitrite by passage through a copperized Cadmium column on channel 1. The resulting Nitrite is

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reacted as above and read at 520 nm. This yields Nitrite plus Nitrate. Nitrate alone is determined by the difference (Nitrate + Nitrite) - Nitrite.

3.0 INTERFERENCES

3.1 A build up of suspended matter in the reduction column can restrict sample flow. Since nitrate-nitrogen is found in a soluble state, all samples are pre-filtered through a 0.45-micron filter.

3.2 High concentrations of iron, copper or other metals may result in a low bias. EDTA is added to the samples to eliminate this interference.

3.3 Samples that contain high concentrations of oil & grease may coat the surface of the cadmium.

3.4 Sample color that absorbs in the photometric range used for analysis also will interfere.

3.5 Samples that have a pH less than 5 or greater than 9 will interfere with the color reagent and shorten the life span of the cadmium column. Samples should be adjusted with either HCl or NH_4OH to bring the sample within a pH range of 5-9.

3.6 Samples that contain chlorine shorten the life span of the cadmium column. Samples should be checked with potassium iodide paper for the presence of chlorine and if present should be treated with sodium thiosulfate until a blue color no longer persists.

4.0 APPARATUS AND MATERIALS

4.1 Lachat Autoanalyzer QC8000, software and autosampler operation manuals

4.2 Nitrate/Nitrite board 10-107-04-1-C with 22.5 cm loop and Cadmium Reduction Column

4.3 Nitrite Board 10-107-04-1-C with 22.5 cm loop

4.4 Proportioning pump

4.5 Analytical Balance, accurate to 0.1 mg

4.6 Volumetric Flasks, Class A

4.7 Class A Pipets

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4.8 Amber Glass Bottles

4.9 pH Paper

4.10 KI (potassium iodide) strips

4.11 0.45u Syringe Filters

4.12 Automatic Pipetters

4.13 Cadmium Reduction Column - Prepared columns are ordered from LACHAT and sent to LACHAT to be re-packed as needed. NOTE: CADMIUM IS VERY TOXIC AND CARCINOGENIC. USE COLUMN WITH CARE.

5.0 REAGENTS

5.1 Ammonium Chloride, buffer - Add about 600 mL of laboratory reagent grade water to a one liter volumetric flask. Add 105 mL of hydrochloric acid, 95 mL of ammonium hydroxide and 1 gram of disodium ethylenediamine tetraacetic acid dihydrate (EDTA). Dilute to mark and invert three times. This is stable for one month.

5.2 Sulfanilamide Color Reagent - Add about 600 mL DI H_2O to a 1 L volumetric flask. Add 100 mL of 85% Phosphoric Acid (concentrated), 40 g sulfanilamide and 1.0 g N-(1-naphthyl)ethylenediamine dihydrochloride (NED). Stir on stir plate until dissolved (approx. 20 minutes). This is stable for 1 month.

5.3 Carrier – laboratory reagent grade water

Standard Solutions

5.4 Nitrite Stock Standard (NO_2), 100 mg/L - Dissolve 0.4926g of sodium nitrite in about 600 mL of laboratory reagent grade water. Dilute to one liter with laboratory reagent grade water and mix thoroughly. Add 1 mL of Chloroform as a preservative.

5.5 Nitrate Stock Standard (NO_3), 100 mg/L - Dissolve 0.6068g of sodium nitrate in about 600 mL of laboratory reagent grade water. Dilute to one liter with laboratory reagent grade water and mix thoroughly. Add 0.5 mL of chloroform as a preservative.

5.6 Nitrite Stock LCS, 304.5 ppm, purchased.

5.7 Nitrate Stock LCS, 225.8 ppm, purchased.

5.8 Using volumetric glassware and calibrated adjustable pipets, prepare the standard dilutions as follows:

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Stock Std (SS) Used	Conc. of Stock Std. (mg/L)	Volume Stock Standard Used (mL)	Final Volume (mL)	Conc. of Std. Dilution (mg/L)	Purpose
NO_2 SS (section 5.4)	100	2.0 1.0 0.5 0.25 0.05	100 100 100 100 100	2.0 NO_2 1.0 NO_2 0.5 NO_2 0.25 NO_2 0.05 NO_2	Calibration Curve
NO_2 SS (5.4) NO_3 SS (5.5)	100 100	0.5 0.5	100 100	0.5 NO_2 0.5 NO_3	CCV-NO_2 CCV-NO_3

5.9 Nitrite Working Stock LCS, 1 mg/L – add 60 mL of laboratory reagent grade water to 100 mL volumetric flask. Add 0.328 mL of Nitrite Stock LCS (5.6) dilute to 100 mL with laboratory reagent grade water and mix thoroughly.

5.10 Nitrate Working Stock LCS, 1mg/L – Add approximately 60 mL of laboratory reagent grade water 100 Ml volumetric flask. Add 0.443 mL of Nitrate LCS (5.7). Dilute to 100 mL with laboratory reagent grade water and mix thoroughly.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be stored at <6 °C, without freezing, until time of analysis. Hold time is 48 hours from time of collection. Samples stored more than 48 hours may be preserved with 2 mL concentrated H_2SO_4 per liter and run as NO_2 plus NO_3 (if requested by client). CAUTION: Samples must not be preserved with mercuric chloride or thiosulfate because this will degrade the cadmium column.

7.0 PROCEDURES

Sample Pretreatment

7.1 Water samples: Samples are prepared for analysis by clearing the sample of debris by syringe filtration through a 0.45 micron filter. Using either conc. HCl or conc. NH_4OH, adjust pH to between 5 and 9, if it is below 5 or above 9. Record adjustment in the analysis run information sheet (Figure 1).

7.2 Solid Samples Extraction – Add an amount of laboratory reagent grade water equal to ten times the weight of the dry sample. This slurry is mixed for ten minutes using a magnetic stirrer. The resulting slurry is allowed to stand or may be centrifuged prior to filtration through 0.45 μm filter. Hold times for analytes of interest should be considered

to commence upon the generation of the extract. The extract is analyzed as an aqueous from this point.

Note: Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for more information on subsampling.

Preparation of Matrix Spike

7.3 To prepare matrix spike (MS) for the analytical batch, use a calibrated Eppendorf or equivalent pipetter. Add 0.025 mL of the 100 mg/L CCV standard for NO_3 (5.5) and the 100 mg/L CCV standard for NO_2 (5.4) to a sample cup containing 5 mL of randomly selected sample, or client specified sample.

Analysis Set Up

7.4 Connect the Nitrite board on channel 2 and the Nitrite/Nitrate board on channel 1. Inspect. Check for any residual coloration or solids in lines. Check that the 520 nm filter is installed. Perform any other routine maintenance as necessary and record in the appropriate logbook (Figure 2). Refer to the LACHAT Maintenance logbook for recommended schedule. Allow colorimeter to warm for 30 minutes (this can take place concurrently with the following).

7.5 Put reagent (buffer, carrier and color reagent) lines in cassettes and raise tension levers to proper level to achieve flow through system. (Refer to Figure 3 for NO_3/NO_2 Manifold Diagram.)

7.6 Prior to connecting column, rinse the boards by placing reagent lines in laboratory reagent grade H_2O and pumping through for a few minutes. Then place lines in proper reagent containers and pump until stable baseline is attained.

7.7 Turn off pump. Connect Cadmium column into NO_2/NO_3 board using care not to introduce air bubbles into the column. (See Figure 4 for manufacturer's additional column installation procedures.) Re-start pump and wait for stable baseline.

Method Selection

7.8 Turn on the computer and log into OMNION FIA. Double click on the OMION FIA icon. Log on using your user name and password. The Omnion Data System will appear. Click on the Flow Injection Analysis instrument icon.

7.9 To open the Nitrate/Nitrite method click on the method icon on the tool bar. Select the method NO2NO3 from the methods list. Click OK.

7.10 To open the NO2NO3 tray template click on the tray icon on the tool bar. The tray table will appear. Only the calibration standards will appear here.

7.11 Prior to entering the sample numbers in the sample ID column, all samples in the batch must be placed in a work group created in KIMS. Refer to SOP CA-762, current revision, for instructions on how to create a work group. Once all samples have been work grouped, the sample numbers can be typed into the sample ID column.

7.12 Type in the laboratory sample identification in the sample ID column. After every set of ten samples a CCV NO_2 / CCV NO_3 / CCB set must be run. Every batch must end with the above QC set.

The following is an example of the analytical sequence:

1	2.0 ppm standard	15	5 Samples
2	1.0 ppm standard	16	CCV NO_3
3	0.5 ppm standard	17	CCV NO_2
4	0.25 ppm standard	18	CCB
5	0.05 ppm standard	19	WG####-# (MS)
6	0.0 ppm standard	20	9 Samples
7	CCVNO_3	21	CCV NO_3
8	CCVNO_2	22	CCV NO_2
9	CCB	23	CCB
10	WG####-# (Blank)	24	6 samples
11	WG####-# (LCS NO_3)	25	CCV NO_3
12	WG####-# (LCS NO_2)	26	CCV NO_2
13	WG####-# (Duplicate)	27	CCB
14	WG####-# (MS)		

NOTE: The state of South Carolina requires a method PQL of 0.02 mg/L for Nitrate and Nitrite. When analyzing samples from South Carolina, a calibration standard spiked at a concentration of 0.02 mg/L must be included in the calibration curve.

7.13 Renumber the cups in sequential order by highlighting the cup # column beginning with cup #1. Click on Tray on the menu bar and select Renumber cups.

7.14 To save this Tray, select File on the menu bar then Save Tray As. Enter a file name. (i.e., NO0902A). Use date and A, B, C, etc. for sequence of trays, with “A” being the first tray of the day.

<u>Calibration and Analysis</u>

7.15 Place the five calibration standards in the calibration racks in the RAS samples in descending order. End the calibration sequence with a blank (laboratory reagent grade water).

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7.16 Utilizing the sequence in the tray table that you have created, load all samples in the sample cups. The sample cups will hold up to 5 mL of sample.

7.17 The tray can now be started. Click on the Tray Run icon showing the yellow arrow that is on the Tool Bar. A window will appear. Click on Catalog next to the data file cell. Another window will appear allowing you to catalog the tray. Select the NO2NO3 Catalog and give the tray a file name. The same file name as above can be used (NO0902A). Click on Run at the bottom of this window. The autosampler will advance to the calibration tubes and begin the calibration. If the calibration passes the autosampler will advance to the sample cups. If the calibration fails the sampler will stop and give you the opportunity to recalibrate. When the tray has finished, the calibration curve (Figure 5) and sample results (Figure 6) can be printed.

7.18 Acceptance criterion is a 0.995 correlation coefficient or better for the full curve for both nitrite and nitrate plus nitrite. It is important to compare curves and absorbances vs. standard concentrations with those previously generated to insure the quality of the data. Printouts of calibrations are filed in the lab for approximately 3 months. Prior calibrations are archived. All are available in database. Significant changes should be investigated. (Switching to fresh standards or fresh reagents may change absorbance slightly.) Check historical data. Operator discretion in approving or rejecting calibrations is encouraged. The decision and rationale should be recorded in the instrument log.

7.19 When a valid calibration is attained (as described in 7.18), the samples can be run.

7.20 The sequence of CCV, CCB, 10 samples is repeated until a CCV or CCB fails or until sequence ends. A CCV and CCB are included at the end of the analytical sequence.

7.21 As samples are running, periodically review sample results, CCVs, CCBs, and raw peak data for any irregularities (e.g., carry over from concentrated samples, poor recoveries, failing calibration verifications, air bubbles, samples requiring dilution, etc.) Note irregularities and QC nonconformances on raw data. Samples with concentrations exceeding the highest calibration standard must be diluted to the midrange of the calibration curve and reanalyzed.

7.22 Analytical results will only be considered as valid data if they fall between acceptable calibration verification/calibration blanks. Any deviations from this must be cleared with the Department Manager, Operations Manager, and/or Quality Assurance Officer.

7.23 When all samples have been run, disconnect column and place lines in water to flush system. Allow system to flush for 5-10 minutes then remove lines from water and allow pump to 'dry' the system. Turn off pump, release tension levers and 'pop' pump cassettes.

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NOTE: The state of South Carolina requires a method PQL of 0.02 mg/L for Nitrate and Nitrite. When analyzing samples from South Carolina, a calibration standard spiked at a concentration of 0.02 mg/L must be included in the calibration curve.

Calculations and Reporting

7.24 Information which will appear on the raw LACHAT run includes: date, analyst, calibration file number, results file number, sample number, dilution, instrument reading, reported concentration, and analysis time. Raw instrument data should clearly indicate QC sample recoveries, reasons for acceptance or rejection of data.

7.25 The cadmium column efficiency should be calculated as follows:

For the ICV/LCS & CCV NO_3 Stds.: $\frac{\text{Observed conc. of } NO_2 + NO_3 \text{ (mg/L)}}{\text{True Value of } NO_3 \text{ standard (mg/L)}} \times 100$

This formula will determine the efficiency of the cadmium column in channel 1 (NO_2 + NO_3) to reduce all of the nitrate standard to nitrite.

The following formulas are used to calculate the NO_2 and NO_3 concentrations:

7.26 Aqueous Samples:

Reported Conc. NO_3 = [(Obs. Conc. NO_2 + NO_3) - Obs. conc. NO_2] X DF

Reported Conc. NO_2 = Obs. Conc. NO_2 X DF

Reported Conc. NO_2 + NO_3 = (Obs. Conc. NO_2 + NO_3) X DF

Where: "DF" is the dilution factor at the instrument
"Obs Conc" is the concentration of the diluted sample

Note: If the Obs. Conc. of NO_2 is greater than the Obs. Conc. of NO_2 + NO_3, the sample should be reanalyzed as is or reanalyzed at dilution.

7.27 Non-Aqueous Distilled Samples (To be applied to all samples extracted by weight):

Reported Conc. NO_2 (mg/Kg) = $\frac{\text{(Rpt. Conc. } NO_2\text{)* Extract Vol (L)}}{\text{Initial Wt.(Kg) * \%TS}}$

Reported Conc. NO_2 + NO_3 (mg/Kg) = $\frac{\text{(Rpt.. Conc. } NO_2 + NO_3\text{)* Extract Vol (L)}}{\text{Initial Wt.(Kg) * \%TS}}$

$$\text{Reported Conc. } NO_3 \text{ (mg/kg)} = \frac{(\text{Rpt. Conc. } NO_3) * \text{Extract Vol (L)}}{\text{Initial Wt.(Kg)} * \%TS}$$

where:

Rpt. Conc. = the concentration of the solution analyzed as determined by the instrument (mg/L) multiplied by the instrument dilution factor.
Ext. Volume = volume of the extract (L) from 7.2
Initial Wt. = initial sample weight (Kg) from 7.2
%TS = percent solids, expressed as a decimal (e.g., 95%=0.95)

7.28 When valid data has been obtained, the prep data can be entered in KIMS. See SOP CA-762, current revision, for this procedure. The LACHAT data can now be exported to KIMS.

7.29 To export data, click on the data icon below the tool bar (green folder). The Open FIA Data File box will appear. Click on Data in the folder box to the right. Select NO2NO3. All of the NO2NO3 files will appear in the box to the left. Select the data file that is needed from this list. Click on File on the tool bar and select Export Data. The Export Data box will appear. Click the OK box. The Save Export Data As box will appear. Highlight the LACHAT file and click OK.

7.30 Once the data is in KIMS the WETREV step can be completed. See SOP CA-762, current revision, for this procedure.

7.31 A batch sheet will print out automatically once the WETREV is done (Figure 7). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.

7.32 All batch sheets, raw data, and supporting documents are scanned after final review and the resulting image files are saved on a Katahdin server for use in data package assembly. Image files of raw data are periodically archived by the laboratory's MIS department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The Department Manager, Operations

Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

Every instance of noncompliant method quality control requires the generation of a non-conformance report describing the problem, suspected cause and final resolution. Non-conformance reports must be signed by the initiator, Department Manager, QA officer, and lab management.

8.1 Immediately following calibration, a Continuing Calibration Verification (CCV) an Initial Calibration Verification/LCS and an Initial Calibration Blank must be run. Separate ICVs/LCSs for NO_2 and NO_3 are run to verify the efficiency of the column to reduce NO_3 to NO_2. Continuing Calibration Verifications and Blanks are run every 10 analytical samples thereafter and at the end of the analytical session.

ICV/LCS/Calibration Verifications must yield 90-110% recovery for method 353.2 and method SM 4500 NO_3 F. Calibration Blanks must be below the method PQL (which for Nitrite/Nitrate is 0.05 mg/l.) (Refer to Table 1 for corrective actions).

8.2 The following QC must be run with each Batch of 20 field samples or per day if < 20 field samples:

- Laboratory Control Sample (LCS) - yielding 90-110% recovery.
- Sample Duplications (1/20) - yielding a RPD of $\leq$ 20% ($\leq$ 10% for 4500NO_3 F). This RPD applies to samples > 3X the PQL. RPD should be <100% for samples <3X the PQL.
- Matrix Spikes (LFM) (1/10) - using sample spiked with a known concentration of nitrite and nitrate yielding 90-110% recovery
- Blanks - Blanks must be less than the method PQL. They are run at a frequency of one per batch of samples.

Refer to Table 1 for corrective actions.

8.3 Calibration verifications falling outside of control limits stop an analytical session. All data must fall between calibration verifications and calibration blanks that are within control limits to be considered valid. If ICV/CCVs and/or ICB/CCBs are out of control, the following actions may be taken:

- Evaluate data to determine whether it is reportable (refer to Table 1 for corrective actions)
- Recalibrate and rerun
- Remake standards and/or reagents and rerun
- Troubleshoot at instrument - check for leaks or blockages - check flow
- Consult with LACHAT Technical Assistance as needed (1-800-247-7613)
- If method remains out of control consult group manager

8.4 Laboratory Control Standards falling out of control limits must be evaluated against the associated samples for the batch. See above and Table 1 for appropriate actions.

8.5 Sample Duplicates out of control limits do not necessarily invalidate data. Refer to Table 1.

8.6 Linear Calibration Range (LCR) – The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. If any verification data exceeds the initial values by ± 10 %, linearity must be reestablished. If any portion of the range is shown to be non-linear, sufficient standards must be used to clearly define the non-linear portion.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method. EPA Method 353.2 states MDLs should be determined every six months, when a new operator begins work, or whenever there is a significant change in the background or instrument response.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory

is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Detection (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of the applicable methods for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Methods for the Determination of Inorganic Substances in Environmental Samples", EPA - 600/R - 93 - 100, August 1993 Rev. 2.

"Standard Methods for the Examination of Water and Wastewater", Method 4500-NO_3 F, 18th Edition, 1992.

QuikChem Method No. 10-107-04-1-C

QuikChem 8000 Software Reference Manual

Operating Manual for QuikChem 8000 AutoAnalyzer

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Current Version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

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Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
EPA 353.2 & SM4500-F Nitrate/ Nitrite	Initial Calibration (including five standards plus blank)	Prior to sample analysis	Linear Regression Correlation Coefficient ≥0.995	(1) Investigate source of problem (2) Recalibrate
	Method blank	One per batch of 20 samples	No analyte detected >PQL	(1) Investigate source of contamination (2) Report all sample results <PQL. (3) Report sample results >10X the blank result and flag results with a "B". (4) Reanalyze all other samples associated with the failing blank.
	LCS/ICV	Immediately after initial calibration and one per batch of 20 samples thereafter.	90-110 %R	(1) If the LCS fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.
	CCV	One after every 10 samples	90-110 %R	(1) If the CCV fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze samples back to last acceptable CCV recovery
	Matrix Spike (LFM)	One for every set of 10 samples	90-110% R	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.
	Sample Duplicate	One sample duplicate per 20 samples	RPD ≤20 (≤10% for 4500NO_3 F). Applies to samples >3X the PQL. RPD should be <100% for samples <3X the PQL.	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still out, report original result with notation or narration.
	LCR	Initially at instrument setup and every six months there after	± 10 % of true value	Reestablish linearity

TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

TABLE 1 (cont.)

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
EPA 353.2 & SM4500-F Nitrate/ Nitrite	Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially and annually thereafter.	Must pass all applicable QC for method	Repeat analysis under supervision until able to perform passing QC; document successful performance in personal training file
	MDL study and/or LOD/LOQ Verifications.	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision. Note- MDL needed every six months.		

TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-728-08	METHOD 353.2 and 4500NO_3 F, current revisions
Apparatus/Materials	1) LACHAT Autoanalyzer with dual column – one with cadmium reduction and one without. 2) 520 nm filters	1) Technicon Autoanalyzer II – one column with cadmium reduction. 2) 353.2: 540 nm filters
Reagents	1) Standard concentrations: 0.05 to 2.00 mg/L of NO_2-N. 2) Color reagent contains 1 g NED 3) Ammonium chloride buffer = 105 mL of HCL, 95 mL of ammonium hydroxide and 1 gram of EDTA to one liter with laboratory reagent grade water	1) 353.2: Standard concentrations: 0.05 to 6.00 mg/L of NO3-N 2) Color reagent contains 2 g NED 3) 353.2: Ammonium chloride solution = 85g of ammonium chloride, 0.1g of EDTA and ammonium hydroxide to pH of 8.5 all brought up to one liter with laboratory reagent grade water. 0.5 mL of Brij-35 (Technicon) is also added. 4500NO_3 F: Ammonium chloride solution = 85g of ammonium chloride diluted to 1L. Add 0.5 mL Brij-35.
Sample preservation/ handling	None.	
Procedures	1) Pre-packed cadmium reduction columns are ordered from LACHAT and are ready to be used 2) Six point curve and calibration verification analyzed every time.	1) Columns are prepared, packed with granulated cadmium and conditioned 2) 353.2 – 3 point curve every time. Expanded curve with three points of calibration verification every six months to confirm linearity.
QC – Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	None	
QC – MDL	None	

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FIGURE 1

EXAMPLE OF NO2/NO3 RUN INFORMATION SHEET

KATAHDIN ANALYTICAL SERVICES, INC.
Wet Chemistry Analysis Run Information Sheet

Analyte: NO3, NO2, and NO3+NO2	**Analyst:** Ra
Instrument: LACHAT	**Analysis Date:** 3/14/12

Were pHs of all samples adjusted to 5-9 prior to analysis?
Circle one: (Yes) No SF1478-8 pH adjusted

Were all samples checked for the presence of chlorine prior to analysis?
Circle one: (Yes) No

Analytical Method (Check all that apply):

☑ EPA 353.2 ☐ SM 4500 F ☐ Other

Reagent Information:

Reagent Name	Reagent ID	Expiration Date
Ammonium Chloride Buffer	W10510	4/14/12
Sulfanilamide Color Reagent	W10493	4/6/12
Carrier	DI Water	

Standards Information:

Standard Name	Concentration (mg/L)	ID	Expiration Date
Nitrate CCV	0.5	W10511	4/14/12
Nitrite CCV	0.5	W10497	4/6/12
Nitrate ICV/LCS	1.0	W10484	3/28/12
Nitrite ICV/LCS	1.0	W10485	3/28/12
Standard #1	2.0	W10497	4/6/12
Standard #2	1.0	↓	↓
Standard #3	0.5	↓	↓
Standard #4	0.25	↓	↓
Standard# 5	0.05	↓	↓
Standard #6	0.0	DI water	N/A
Nitrate Standard	100.0	W10133	8/3/12
Nitrite Standard	100.0	W9986	6/27/12

Notes:

1. Matrix Spiking: To 5mL Sample Alliquot add 0.025 mL of Nitrate Standard and 0.025mL Nitrite Standard

Comments:

TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

FIGURE 2

EXAMPLE LACHAT MAINTENANCE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC. – WET CHEMISTRY SECTION
LACHAT Quickchem 8000 Ion Analyzer – Serial No. A83000-1642

DAILY INSTRUMENT MAINTENANCE

Task	Enter Dates Performed and Initials of Analyst Below					
Clean instrument, pump, autosampler, and detector surfaces	9/27/11 CT	9/28/11 CT	CT 9/29/11	CT 10/1/11		
Clean counter around Instrument						
Check waste container, empty as necessary	↓	↓	↓	↓		
Rinse pump cartridges						

ADDITIONAL INSTRUMENT MAINTENANCE

Maintenance Frequency	Task	Date / Initial Last Performed
Weekly	Clean injection port and valve connectors	10/1/11
Monthly	Clean pump rollers with silicone spray	10/1/11
	Clean autosampler rods and moving parts	10/1/11
	Check pump tubes and replace as needed	ND,ND,10/1/11
	Clean unions and tees	ND,ND,10/1/11
Every Six Months	Replace all manifold and injection valve O-rings	
	Clean hard drive on computer	
Annually	Replace all manifold tubing	
	Replace flow cell flares and O-rings	
	Replace waste tubing	

Comments and Non-Routine Maintenance:
9/27/11 replaced cadmium column

WL-018 – Revision 2 – 07/19/2011

QAWL618

0000012

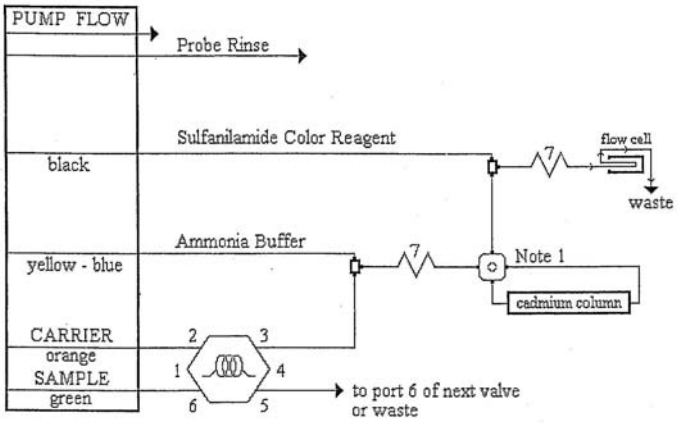
TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

FIGURE 3

NO3/NO2 MANIFOLD DIAGRAM

17. TABLE, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1. NITRATE/NITRITE MANIFOLD DIAGRAM



Sample Loop = 17 cm X 0.8 mm
QC8000 Sample Loop = 22.5 cm X 0.8 mm

Interference Filter = 520 nm

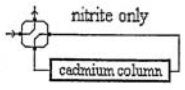
CARRIER is Helium Degassed DI water

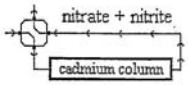
Manifold tubing is **0.8 mm (0.032 in)** i.d. This is 5.2 uL/cm.

7 is **135** cm of tubing on a 7 cm coil support

APPARATUS: An injection valve, a 10 mm path length flow cell , and a colorimetric detector module is required..

Note 1: This is a 2 state switching valve used to place the cadmium column in-line with the manifold.





TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

FIGURE 4

LACHAT CADMIUM COLUMN INSTALLATION PROCEDURE

LACHAT
A Hach Company Brand

50237A-89

Instruction Sheet

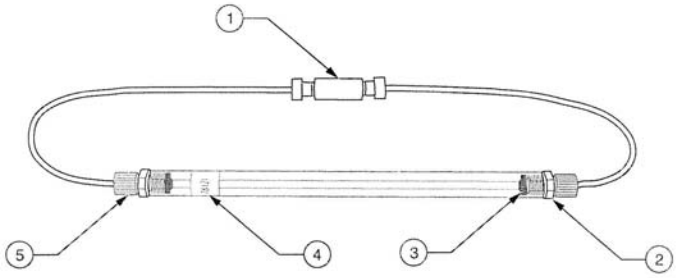
Cadmium Column for Nitrate Determination

Please read this entire document before unpacking, setting up, or operating this instrument. Do not use or install this equipment in any manner other than that which is specified in this instruction sheet.

The new Cadmium Column for Nitrate Determination is a disposable alternative to repackable cadmium columns.

For disposal instructions, see Disposal of Used Cadmium Columns on page 4.

Figure 1 Cadmium Column



1. Union	2. Jam Nut	3. Column Seal	4. Lot Number	5. Flangeless Fitting (2)

Inspection

After unpacking the cadmium column, visually inspect for:

- Gaps in the column
- Changes in cadmium granule color (should be gray)
- Changes in the cadmium surface characteristics

If any of these problems are present, be sure to check the column officiency before running samples.

Installation

Before installing the column, pump all reagents through the manifold. Make sure the switching valve is off-line.

1. Connect a union fitting between the two detached flares at the switching valve (see Figure 2).
2. Turn the switching valve to the in-line position for about 5 seconds, then return it to the off-line position. This will ensure that buffer solution is present in the flared tubing.
3. Remove the flared tubing from one side of the union fitting.
4. Disconnect the Teflon®* tubing from one side of the union fitting attached to the cadmium column.

TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

FIGURE 4 (cont.)

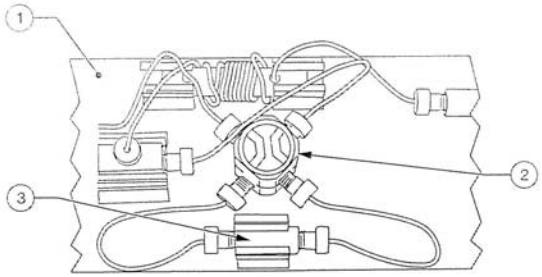
LACHAT CADMIUM COLUMN INSTALLATION PROCEDURE

Cadmium Column for Nitrate Determination

5. Connect the cadmium column to the switching valve as shown in Figure 3. The direction of the column is irrelevant.
6. Turn the switching valve to the in-line position before starting the analysis.

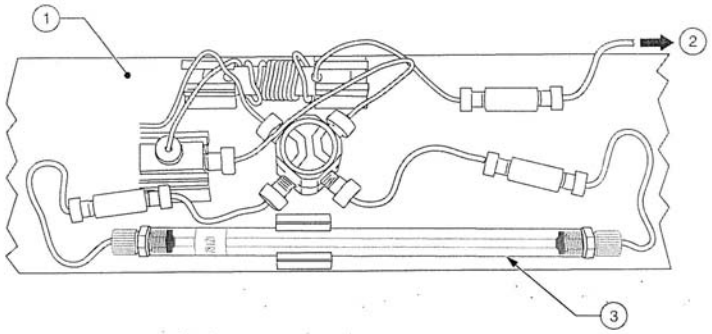
Note: Samples with pH less than 1.0 and high oil content could damage the cadmium column. These samples should be pretreated before running them through the cadmium column.

Figure 2 Switching Valve



1. Nitrate Manifold	2. Switching Valve	3. Union Fitting

Figure 3 **Switching Valve**



1. Nitrate Manifold	2. Tubing to Flow Cell	3. Cadmium Column

*Teflon is a registered trademark of DuPont Corporation

TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

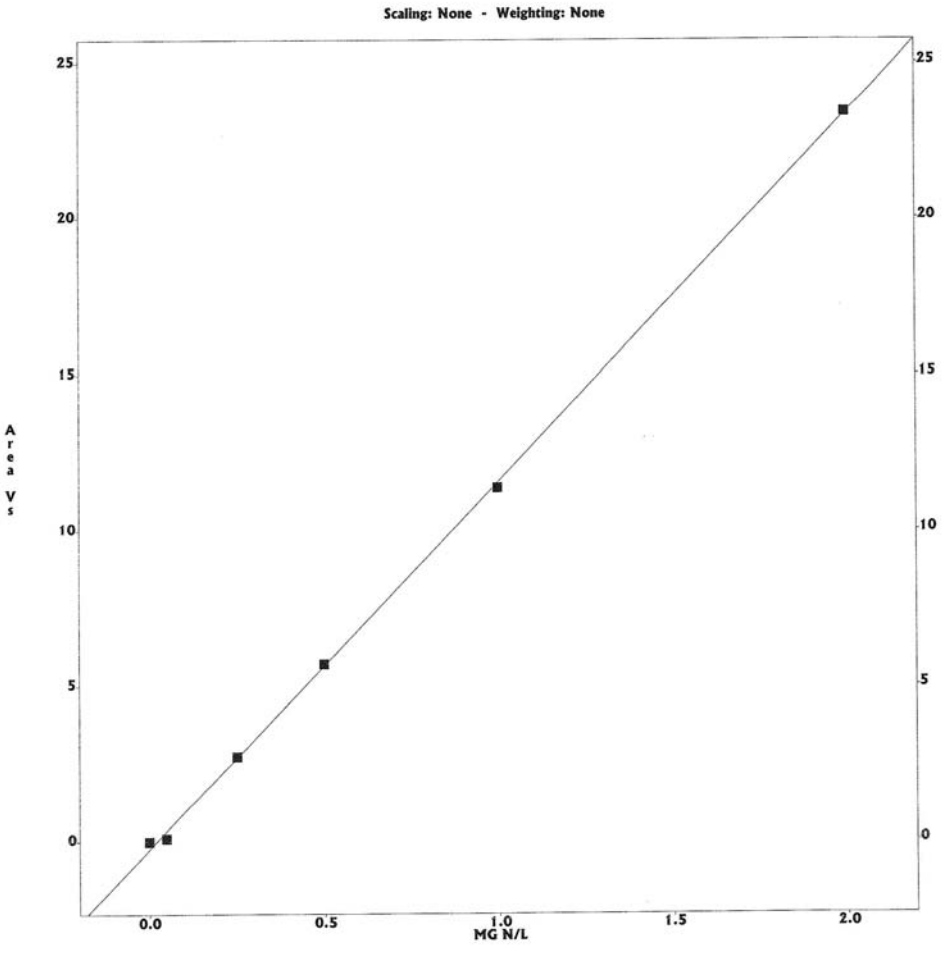
FIGURE 5-A

EXAMPLE OF NO3/NO2 CALIBRATION CURVE

NITRATE/NITRITE

Lvl	Area	MG N/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	23404966	2.00	23404966					0.0	0.0	-0.3
2	11383069	1.00	11383069					0.0	0.0	1.4
3	5705206	0.50	5705206					0.0	0.0	-1.0
4	2733709	0.25	2733709					0.0	0.0	-1.2
5	101444	0.05	101444					0.0	0.0	40.5
6	0	0.00	0					0.0	0.0	

1st Order Poly
Conc = 8.478e-008 Area + 2.113e-002
r = 0.9998



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TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

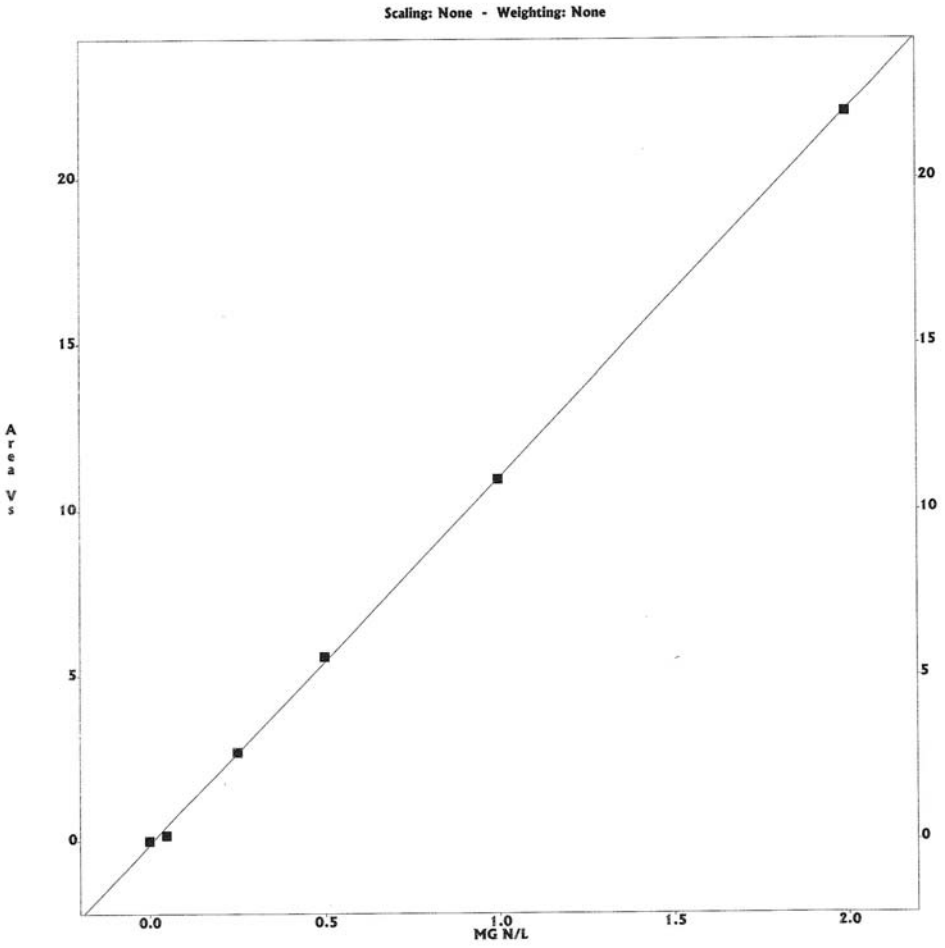
FIGURE 5-B

EXAMPLE OF NO2 CALIBRATION CURVE

NITRITE

Lvl	Area	MG N/L	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	22019326	2.00	22019326					0.0	0.0	0.2
2	10954059	1.00	10954059					0.0	0.0	0.1
3	5586276	0.50	5586276					0.0	0.0	-3.0
4	2684570	0.25	2684570					0.0	0.0	-1.2
5	161330	0.05	161330					0.0	0.0	48.8
6	0	0.00	0					0.0	0.0	

1st Order Poly
Conc = 9.017e-008 Area + 1.104e-002
r = 0.9998



Printed: Friday, March 16, 2012 - 11:36 AM

TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

FIGURE 6

EXAMPLE OF NO3/NO2 SAMPLE PRINTOUT

OPERATOR: RO
ACQ. TIME: Mar 16, 2012 11:05:46
DATA FILENAME: C:\OMNION\DATA\NO2NO3\NO03162A.FDT
METHOD FILENAME: C:\OMNION\METHODS\NO2NO3.MET
TRAY FILENAME: C:\OMNION\DATA\NO2NO3\NO03162A.TRA

WG105954
R190015

Multi-Channel Table
Type: Unknowns
Channel Range: 1 to 8 -- Cup Range: 1 to 50

Cup	Sample ID	Sampling Date	Sampling Time	# of Reps	NITRATE/NITRITE (MG N/L)		NITRITE (MG N/L)		Man Dil Factor
1	CCVNO3	16 Mar 2012	11:13:13	1	0.4865	97.3	0.0110		1.0
2	CCVNO2	16 Mar 2012	11:14:16	1	0.4996		0.5144	102.8	1.0
3	CCB	16 Mar 2012	11:15:20	1	0.0211		0.0110		1.0
4	WG-1	16 Mar 2012	11:16:23	1	0.0211		0.0110		1.0
5	WG-2	16 Mar 2012	11:17:26	1	0.8989	Wrong WG#	0.0110		1.0
6	WG-3	16 Mar 2012	11:18:29	1	0.9800		1.0057		1.0
7	MW102	16 Mar 2012	11:19:31	1	0.0211		0.0110		1.0
8	WG-4	16 Mar 2012	11:20:34	1	0.0211		0.0110		1.0
9	WG-5	16 Mar 2012	11:21:36	1	1.0915		0.6185		1.0
10	MW102S	16 Mar 2012	11:22:39	1	0.0287		0.0329		1.0
11	MW103	16 Mar 2012	11:23:41	1	0.0930	pH adjusted	0.0110		1.0
12	MW103S	16 Mar 2012	11:24:44	1	0.0293		0.0263		1.0
13	MW902	16 Mar 2012	11:25:45	1	0.4701		0.0177		1.0
14	CCVNO3	16 Mar 2012	11:26:47	1	0.4707	94.14	0.0110		1.0
15	CCVNO2	16 Mar 2012	11:27:48	1	0.4888		0.5178	103.56	1.0
16	CCB	16 Mar 2012	11:28:52	1	0.0211		0.0110		1.0
17	DUP01	16 Mar 2012	11:29:56	1	0.0318		0.0273		1.0
18	CCVNO3	16 Mar 2012	11:30:59	1	0.4631	92.62	0.0110		1.0
19	CCVNO2	16 Mar 2012	11:32:03	1	0.4822		0.5152	103.04	1.0
20	CCB	16 Mar 2012	11:33:06	1	0.0211		0.0110		1.0

TITLE: **TOTAL NITRATE/NITRITE, NITRITE & NITRATE WITH CADMIUM REDUCTION BY AUTOMATED COLORIMETRY**

FIGURE 7

EXAMPLE OF NO3/NO2 BATCH SHEET

WET CHEMISTRY BATCH REPORT
Mar 19 2012, 10:06 am
Batch: WG105954

Parameter: Nitrate As N
Prep Date: N/A

Date Analyzed: 16-MAR-12
Prep Method: N/A

Analyst Initials: RO
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SF1478-10	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	.0053	U0.025 mg/L	NA	.05	.0152	0.050		
SF1478-13	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	.4419	0.44 mg/L	NA	.05	.0152	0.050		
SF1478-2	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	.0101	U0.025 mg/L	NA	.05	.0152	0.050		
SF1478-3	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	-.0042	U0.025 mg/L	NA	.05	.0152	0.050		
SF1478-4	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	.003	U0.025 mg/L	NA	.05	.0152	0.050		
SF1478-5	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	.0105	U0.025 mg/L	NA	.05	.0152	0.050		
SF1478-6	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	.0045	U0.025 mg/L	NA	.05	.0152	0.050		
SF1478-7	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	.4523	0.45 mg/L	NA	.05	.0152	0.050		
SF1478-8	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	.082	0.082 mg/L	NA	.05	.0152	0.050		
SF1478-9	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	1.8371	1.8 mg/L	NA	.05	.0152	0.050		
SF1495-1	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	.0101	U0.050 mg/L	NA	.05	0.0040	0.050		
SF1498-1	SAMP	EPA 353.2	5.0000mL	5.0000mL	1	.62	0.62 mg/L	NA	.05	0.0040	0.050		
WG105954-1	MBLANK	EPA 353.2	5.0000mL	5.0000mL	1	.0101	U0.025 mg/L	NA	.05	.0152	0.050		
WG105954-2	LCS	EPA 353.2	5.0000mL	5.0000mL	1	.9426	0.94 mg/L	NA	.05	.0152	0.050		94
WG105954-4	DUP	EPA 353.2	5.0000mL	5.0000mL	1	.0101	U0.050 mg/L	NA	.05	.0152	0.050	NC	
WG105954-5	MS	EPA 353.2	5.0000mL	5.0000mL	1	.4731	0.47 mg/L	NA	.05	.0152	0.050		95
WG105954-6	DUP	EPA 353.2	5.0000mL	5.0000mL	1	1.8124	1.8 mg/L	NA	.05	.0152	0.050	1	
WG105954-7	MS	EPA 353.2	5.0000mL	5.0000mL	2	2.2531	2.2 mg/L	NA	.05	0.030	0.10		83

Comments:

SF1478-10	DOD
SF1478-13	DOD
SF1478-2	DOD
SF1478-3	DOD VOA is unpres.
SF1478-4	DOD
SF1478-5	DOD
SF1478-6	DOD VOA is unpres.
SF1478-7	DOD
SF1478-8	DOD
SF1478-9	DOD MS/MSD
WG105954-1	SF1478-2
WG105954-2	SF1478-2
WG105954-3	SF1478-2
WG105954-4	SF1478-2
WG105954-5	SF1478-2
WG105954-6	SF1478-9
WG105954-7	SF1478-9

Entered by: RO Date: 3/16/12 Accepted by: [signature] Date: 03/19/12

TITLE: **ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056**

Prepared By: Deborah McGrath Date: 05/00

Approved By:

Group Supervisor: Keith Tangley Date: 052901

Operations Manager: J. Burton Date: 5/23/01

QA Officer: Deborah J. Nadeau Date: 5.22.01

General Manager: Deborah F. Nadeau Date: 5/24/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02	Format changes, added pollution prevention, added SW846 method 9056 reference and requirements. Updated logbook attachments.	DN	5.22.01	
03	Added reference to KIMS data entry (Sect. 7.14). Replaced figs 2 & 5. added wording to sect. 8. minor changes throughout to reflect current practices	LAD	031805	031805
04	Added information on Retention time Windows. Added reference for SOP CA 107 to Section 1.4	LAD	04/06	04/06
05	Sect. 5.0 - edited to reflect current Std. prep procedures. Sect. 7.0 - changed base range, added syringe purging procedure, added info for setting up calibration in active sequence, added need for new sequence at beginning of every month	LAD	06/08	06/08
06	Minor revisions to sections 1.1 and 4 for clarity. Revisions to Section 8, 10, Table 2 and 3 to update from SW846 method 9056 to 9056A. Added LLQQ criteria to Section 8.	LAD	02/09	02/09

TITLE: **ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Sect. 1.1- Added definitions. Sect. 4.1- Added computer and software references. Sect. 8, Table 1 and Table 2 - added DoDQSM requirements. Sect. 10. Added references.	LAD	08/09	08/09
08	Added creating and viewing audit log for any result or method file which has been edited. Added Table 2- DoDQSM QC Criteria.	LAD	10/10	10/10
09	Sect. 4, 5 and 7 - updated throughout for new instrument and the addition of Fluoride. Sect. 9- Added MDL, LOD and LOQ information Sect. 10- Added & edited references. Updated Fig. 1 → 3 & 5 (renamed 4). Removed Fig. 4 & 6 (Turbochrom examples).	LAD	06/12	06/12
10	Sect. 5 - updated std. concs. Sect. 7 - Removed information relating to old instrument. Table 3 - Added DODQSM 5.0 QC criteria. Renumbered subsequent Tables. Changed KASINC to KAS throughout.	LAD	08/15	08/15

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-742-10,** titled **ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

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Recipient: _____ Date: _____

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by Katahdin Analytical Services technical personnel to determine the concentration of the following inorganic anions using ion chromatography (IC) by EPA Method 300.0 and SW-846 9056, current version: Sulfate, Bromide, Phosphate-P, Chloride, Fluoride, Nitrate-N, and Nitrite-N. This method may be used for the analysis of the following matrices: Drinking water, Surface water, Mixed Domestic and Industrial Waste waters, Ground water, Reagent waters, solids (after aqueous extraction), and Leachates (when no acetic acid is used).

1.1 Definitions

Analytical Batch – A group of 20 or fewer samples that are analyzed together on the same day.

Calibration Blank (CB) - A volume of laboratory reagent grade water fortified with the same matrix as the calibration standards but without the analytes. In most cases the CB will consist of laboratory reagent grade water.

Continuing Calibration Blank (CCB) – An aliquot of reagent water that is analyzed after each CCV to ensure continuing calibration accuracy.

Continuing Calibration Verification (CCV) – A midrange standard containing all method analytes that is run at the beginning of each run, after every 10 samples, and at the end of each run to ensure continuing calibration accuracy. The CCV is prepared from the same source as the calibration standards. The CCV is sometimes called the Instrument Performance Check Solution (IPC).

Laboratory Control Sample (LCS) / Initial Calibration Verification (ICV) – An aliquot of reagent water to which known amounts of the method analytes are added, and that is processed through the entire analytical procedure in the same manner as a sample. One LCS is processed and analyzed with each batch of 20 or fewer samples. The LCS/ICV is prepared from a different standard source than the calibration standards. LCSs provide a sample of known concentration to assess the accuracy of the analytical system, and when analyzed in duplicate may be used to a measure of precision for the analytical system. The LCS/ICV is sometimes called the Laboratory Fortified Blank (LFB).

Laboratory Duplicate – A duplicate is a second aliquot of a sample that is analyzed to to assess the precision of the analysis.

LOD – Limit of Detection. The smallest amount or concentration of an analyte that must be present in a sample to be detected at a 99% confidence level. At the LOD, the false negative rate is 1%.

Matrix Spike (MS) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for the background concentrations. The MS is sometimes called the Laboratory Fortified Matrix (LFM).

Method Blank (MB) – An aliquot of reagent water that is carried through the entire analytical procedure in the same manner as a sample. One method blank is processed and analyzed with each batch of 20 or fewer samples.

Method Detection Limit (MDL) – The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

Practical Quantitation Limit (PQL) – The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the MDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of anions by IC using EPA Method 300.0 and SW-846 9056. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of anions by IC using EPA Method 300.0 or SW-846 9056 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab logbook. Any deviations from the test or irregularities with the samples should also be recorded in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to

all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

The outflow from the chromatograph and the autosampler is collected in a single container and disposed of in a Satellite Waste “N-HI” Acid for proper disposal in main waste area “A”. Other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOPs SD-903, “Sample Disposal” and CA-107, “The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and Standards,” current revisions. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

A small volume of sample is introduced into an ion chromatograph. The anions of interest are separated and measured using a system comprised of a guard column, analytical column, suppressor device and conductivity detector. An aqueous extraction procedure must be performed in order to utilize this method for solid matrices.

3.0 INTERFERENCES

3.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems. The most common source of interference is high chloride concentrations.

3.2 The water dip or negative peak that elutes near and can interfere with the chloride peak can usually be eliminated by the adding the equivalent of 1mL of eluent concentrate to 100 mL of each sample.

3.3 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

3.4 Method interferences may be caused by contaminants in the water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in the ion chromatograph.

3.5 Samples that contain particles larger than 0.45 μ and reagent solutions that contain particles larger than 0.2 μm require filtration to prevent damage to instrument columns and flow systems.

3.6 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations above 1.5 mg/L fluoride this interference may not be significant.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph capable of delivering 1 to 5 mL of eluent per minute at a pressure of 1000 to 4000 psi. The chromatograph is controlled with a Windows-based PC running Chromeleon software. The chromatograph must be equipped with an injection valve, a 10- to 100-uL sample loop, and set up with the following components.

4.1.1 Autosampler – Dionex Model AS-DV

4.1.2 Ion chromatography system – Dionex ICS-2000 with conductivity detection

4.1.3 Precolumn – A guard column placed before the separator column to protect the separator column from fouling by particulates or organic constituents. Dionex Guard Cartridge AG18 or equivalent.

4.1.4 Separator column – A column packed with an anion exchange resin, suitable for resolving the anions of interest. Dionex Ionpac AS18 or equivalent.

4.1.5 Conductivity suppressor – An ion exchange-based device that is capable of converting the eluent and separated anions into their respective acid forms. Dionex Micromembrane Suppressor Model ASRS 300 or equivalent.

4.2 Analytical balance capable of accurately weighing to the nearest 0.0001g.

4.3 0.5 mL sample vials and filter caps

4.4 Eppendorf pipets, assorted volumes

4.5 Class "A" volumetric flasks, assorted volumes

4.6 Chromeleon software

5.0 REAGENTS

5.1 Eluent Generator Cartridge, Potassium Hydroxide

5.2 Laboratory reagent water

5.3 Stock Standard Solutions: Stock standard solutions may be purchased as certified solutions or prepared from ACS reagent grade material. All purchased standards prepared from high purity salts and supplied by the vendors with certificates of purity and analysis. All purchased stock standards are given an expiration date as indicated by the manufacturer.

5.3.1 Bromide (Br^-), 1000 mg/L, purchased.

5.3.2 Chloride (Cl^-), 1000 mg/L, purchased

5.3.3 Nitrate (NO_3^--N), 225.9 mg/L as N (1000 mg/L as NO_3), purchased

5.3.4 Nitrite (NO_2^--N), 304.4 mg/L as N (1000 mg/L as NO_2), purchased

5.3.5 Phosphate (PO_4^{-3}-P), 1000 mg/L as P, purchased

5.3.6 Sulfate (SO_4^{-2}), 1000 mg/L, purchased

5.3.7 Fluoride (F^-), 1000 mg/L, purchased

5.4 Initial Calibration Stock Standard Mix- Combine the following and dilute to 200 mL with laboratory reagent grade water:

Analyte	Amount of Stock Std. Added (mL)	Concen-tration in Stock Std. (mg/L)	Final Volume of Primary Mixed Cal. Std. (mL)	Final Conc. In Primary Mixed Cal. Std. (mg/L)
Cl^-	2.0	1000	200	10.0
NO_2^--N	2.63	304.4		4.0
NO_3^--N	3.54	225.9		4.0
Br^-	4.0	1000		20.0
SO_4^{-2}	4.0	1000		20.0
PO_4^{-3}-P	1.0	1000		5.0
F^-	1.0	1000		5.0

NOTE: At any time a stock may be prepared with an abbreviated list of analytes and should be clearly labeled with the list of analytes contained.

5.5 Initial Calibration Working Standards – Dilute Initial Calibration Stock Standard as follows to prepare the five-point level working calibration standards:

Work-ing Std. ID	Amount of Primary Mixed Cal. Std. Added (mL)	Final Volume of Working Cal. Std. (mL)	Analyte Conc. In Working Cal. Std. (mg/L)						
			Cl^-	F^-	NO_2^- as N	NO_3^- as N	Br^-	SO_4^{-2}	PO_4^{-3} as P
#6	1	1	10	5	4	4	20	20	5
#5	0.5	1	5	2.5	2	2	10	10	2.5
#4	0.25	1	2.5	1.25	1	1	5	5	1.25
#3	0.1	1	1	0.5	0.4	0.4	2	2	0.5
#2	0.01	1	.1	0.05	0.04	0.04	0.2	0.2	0.05

Note: Standard #1 is the calibration blank

5.6 Continuing Calibration Verification Standard (CCV) – The CCV will be prepared using the same stock as the calibration standards and will be prepared at the same concentration as calibration standard #5.

5.7 LCS/MS Stock Standard – LCS/MS Stock must be comprised of independent sources for all analytes relative to the calibration standard stock. Combine the following using purchased standards from second source and dilute to 200 mL with laboratory reagent grade water: 7.5 mL of standard solution “A” and 7.5 mL of standard solution “B”.

5.7.1 Standard Solution "A"- Multi-element standard which contains analytes in the following concentrations:

- Nitrate (as N) – 225.9 mg/L
- Bromide- 1000 mg/L
- Orthophosphate (as P)- 326.1 mg/L
- Chloride – 1000 mg/L
- Fluoride – 1000 mg/L
- Sulfate- 1000 mg/L

5.7.2 Standard Solution "B" – Contains Nitrite (as N) only in the concentration of 304.4 mg

5.8 LCS Working Standard – The Working LCS is made by adding 0.05 mL of LCS Stock Standard to 0.950 mL of DI water for a final volume of 1.00 mL. This will yield analyte concentrations as follows:

F	Cl	NO2 (as N)	NO3 (as N)	Br	SO4	PO4 (as P)
3.75	3.75	1.14	.845	3.75	3.75	1.22

5.9 Matrix Spiked Sample – A spiked sample aliquot is prepared by adding 0.05 mL of Stock Standard (5.9) to 0.950 mL of sample for a final volume of 1.00 mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean glass or polyethylene bottles. Sample preservation and holding times for the anions that can be determined by this method are as follow.

ANALYTE	PRESERVATION	HOLDING TIME
Bromide	None required	28 days
Chloride	None required	28 days
Nitrate-N	Cool to 4^{o}C	48 hrs
Nitrite-N	Cool to 4^{o}C	48 hrs
Ortho-Phosphate-P	Cool to 4^{o}C	48 hrs
Sulfate	Cool to 4^{o}C	28 days
Fluoride	Cool to 4^{o}C	28 days

NOTE: Due to the potential of and persistence of chloride contamination and the associated short hold times for nitrite, nitrate and ortho-phosphate, it is recommended that these ions are run concurrently by alternate methods.

7.0 PROCEDURES

7.1 SAMPLE PREPARATION

Refer to Katahdin Analytical Services SOP CA-106, "Basic Laboratory Technique", current revision for information on sub-sampling.

7.1.1 Solid Samples Extraction – Add an amount of laboratory reagent grade water equal to ten times the weight of the dry sample. This slurry is mixed for ten minutes using a magnetic stirrer. The resulting slurry is allowed to stand or may be centrifuged prior to filtration through 0.45 μm filter. Hold times for analytes of interest should be considered to commence upon the generation of the extract.

7.2 TURNING ON INSTRUMENT

7.2.1 The instrument is configured as illustrated in Figure 1.

7.2.2 Open the chromeleon software

7.2.3 Click on the instrument tab in the lower left corner.

7.2.4 Click on buttons for pump, Eluent Generator, CR-TC, then suppressor, in that order. The eluent should be set at 30mM, the suppressor should be set at 23mA, and the flow rate should be set at 0.30mL/min. The eluent fill level should be adjusted whenever water is added to the bottle. The column heater should be set at 30^{o} C. The run time is set in the instrument method for 16 min.

Note: If the water has been changed or the instrument has been off for a few days, the pump should be primed before turning the instrument on for the day. This is done by clicking the prime button on the instrument panel, opening the waste valve, and then clicking OK at the top of the screen. Allow to prime for five minutes or so before clicking the off button and closing the valve.

7.2.5 Monitor and record the backpressure on the HPLC pump. The pressure should
not exceed 3000 psi. If this occurs it would indicate the need to replace or clean the column, see column care and maintenance in the column manual.

7.3 CALIBRATION

7.3.1 A new calibration curve must be prepared at least one time every 6 months (daily for SW846 9056) or if one of the following occurs:

7.3.1.1 The daily calibration verification is outside of the method criteria (Refer to section 8.0).

7.3.1.2 Major maintenance has been performed on the instrument (Refer to maintenance log, Figure 2).

NOTE: If one of the above is true, a new calibration curve must be prepared even if it has been less than 6 months since the last calibration.

7.3.2 If a calibration curve is required, prepare the standards as described in section 5.0. Pipet 1 mL of each standard into the autosampler cups and push filter caps down into the vials using the filter cap tool.

7.3.3 Record the standards in the runlog (Figure 3) and load onto the autosampler. Push the carousel release button on the autosampler to allow the wheel to turn. Push the button again to engage the wheel. To start a new sequence, open an old anion sequence in chromeleon by clicking on the data tab and double clicking the desired sequence. Old samples can be deleted by row. Click “save as” and use that day’s date as the sequence name. When entering the calibration points into the sample sequence, change the sample type to “calibration standard” and type the calibrator names in the level column. Two method blanks that will not be reported should be analyzed prior to calibration or sample run to ensure that there is no contamination present in the system. The instrument method and the processing method should not need to be changed. The volume column should read 25 uL for all samples. The “fill down” button can be clicked to renumber the position column. Resave the sequence and click the start button at the top of the screen.

7.3.4 When the calibration is finished, the calibration will need to be updated in the processing method. This is done by double clicking on the processing method on the bottom of the data screen in the associated items table. The calibration tab should already be selected. Click the browse button in the global calibration settings box. Double click the desired sequence, then click update. Save the processing method. The chromatograms for the entire sequence can be printed by double clicking on any chromatogram in the ECD_1 column and clicking on report designer in the lower left corner. Click on the integration tab at the bottom of the screen, then click the chromeleon icon in the upper left to print. Click print and check apply to current sequence. Click OK.

7.3.5 To print out all of the calibration curve graphs highlight all of the points in the sequence and right click to select print. Click the button next to report template and select the method folder. Double click on “test” and deselect all but the calibration box. Click OK to print.

7.3.6 The acceptance criteria for the calibration is a 0.995 correlation coefficient for the full curve. Also, the standard at or below the PQL must be within 50% of

the true value (for SW 9056A only). It is important to compare curves and responses vs. standard concentration with those previously generated to insure quality of data. Significant changes should be investigated. Switching to fresh standards or fresh reagents may change response slightly. Check historical data. Operator discretion in approving or rejecting calibrations is encouraged. The decision and rationale should be recorded in the logbook.

7.3.7 Retention time windows should be established when a new column is installed; if there is a change in instrument conditions or at least annually if there are no changes. Retention time windows are established using +/- three times the standard deviation of the retention times of standards run over the course of one day. The experience of the analyst should weigh heavily in the interpretation of chromatograms.

7.4 LOADING SAMPLES

7.4.1 Write sequence in IC Run Log; follow page format and proper sample coding.

7.4.2 If high sample concentrations are suspected, steps should be taken to minimize reruns and protect the system from contamination and/or carryover. In general samples from potable sources, drinking water samples, may be analyzed at an as received concentration. Monitoring wells, leachates, and estuarine samples may have extremely high concentrations of chloride and/or sulfate. To avoid contaminating the system, analyze these samples at an initial dilution. In the event that the analyzed aliquot does have higher concentrations, inject water samples after the sample to clean out the system. For highly concentrated samples it may take as many as 5 or more water injections to clean out the injector and remove the carryover.

7.4.3 It is recommended that the tray is initially set up to run the opening QC before loading samples. Opening QC consists of a CCV, CCB, Blank, and LCS. If there is no calibration being run, two Blanks must be run at the beginning of the sequence. Evaluate the opening QC to ensure adequate separation, good chromatography, acceptable recovery as it relates to the calibration, and clean blanks. If the opening QC is within criteria and the chromatographic system is in control proceed to load samples. If QC criteria are not met or chromatography is not acceptable initiate appropriate corrective action.

7.4.4 To automatically run tray:

- Press the carousel release button to load the sample wheel. Load samples and press button again to enable the carousel.

- Enter samples into the sequence, filling in all dilutions and positions.
- Make sure the injection volume is 25 uL

- Click the "save" icon and press start. Note: if some samples have already been run, you will need to click "remove" and then "resume" at the top of the screen to continue run.

7.5 SHUT DOWN PROCEDURE – **It is CRITICAL to explicitly follow long term shutdown storage for columns to prevent damage.**

7.5.1 Turn off the instrument in the reverse order from how it was turned on.

7.5.2 **If the system will not be used for more than a week it is critical to fill the columns with the appropriate storage solution and cap them to prevent evaporation. If a column dries out it is most likely useless.**

7.5.3 **It is critical to fill the suppressor with fresh regenerant and cap it. If the suppressor dries out it is likely to be ruined.**

7.5.4 It is best to run the instrument for a little while every couple of days to keep everything hydrated.

DATA ANALYSIS, CALCULATIONS & REPORTING

7.6 If a sample is run and the analyte of interest concentration is above the calibration range the sample must be diluted and reanalyzed. Multiple dilutions may be required to obtain results for all analytes of interest in the calibration range. For samples run at multiple dilutions, the analysis of greatest concentration, qualified retention time, good peak shape, and satisfactory resolution should be quantitated and reported. However, all dilutions should be assessed comparing the consistency of the determinations and possible matrix effects. In certain instances, the more diluted analysis may be the more appropriate reported result.

7.7 If a sample is run at dilution and the concentration of the analyte of interest is below the PQL the sample should be reanalyzed at a greater concentration. Where there is coelution of higher relative concentration analytes to adjacent analytes, e.g. chloride to nitrite, a sufficient dilution of the sample should be analyzed to maximize the resolution of the two analytes and report the affected analyte concentration at an elevated PQL narrated to that effect.

7.8 If the chromatogram fails to produce adequate resolution, or if the identification of specific anions is questionable, the sample may be spiked with an appropriate amount of standard and reanalyzed. In some instances dilution of the sample may provide sufficient resolution for identification and quantitation.

NOTE: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

7.9 Calculations are performed by Chromeleon using responses measured during analysis of the calibration standards for the operating curve that has been calculated based upon a linear regression formula. Individual calibration curves are calculated for each detector. The analyst must assure that the method file is calculating against the appropriate curve. Aqueous and soil sample analyte concentrations are calculated using the following equation:

$$A = (mR + B) * DF$$

where: A = analyte concentration mg/L
m = slope
R = response in peak area
B = y intercept
DF = dilution factor prior to analysis

NOTE: In order for the dilution factor to be applied during calculation by Chromeleon it must be entered at the time the sequence is entered and/or edited.

7.10 In the event that the software interpretation of integration is not appropriate manual integration may be performed. Refer to the Chromeleon software manual for integration techniques. It is expected that the same sound technical judgments and assessments will be equally applied to both samples and standards in the review of integrations and the decisions to perform or not perform manual integrations. In accordance with Section 7.7 of the Katahdin Analytical Services Quality Assurance Manual, any manual integration must be initialed and dated by both the analyst performing the integration and by the reviewer. **Under no circumstances is the original software generated result file to be overwritten with a manually edited file. Both the original software generated integration and the manual integration must be preserved with the raw data.** The analyst should rarely be required to manually integrate any QC elements. This is usually indicative of poor system performance and corrective action should be taken through proper maintenance.

7.11 When analyzing samples using the Ion Chromatograph in the Wet Chemistry Laboratory, the audit log function must be used in order to electronically document the integrity of the data on this instrument. When a change is made to a method or sample file, the user must save the file with the same file number appended with the next letter (i.e., a, b, c, etc.). The audit log for each sample is always available. Click on the sample and then click on the audit trail tab at the bottom of the screen. This will show every step that the sample has gone through.

7.12 The final raw data report will include information on initial and continuing calibrations and results of all quality control data. The instrument printout includes Result File Name, Calibration File Name, Sample #, Analyst, Date Analyzed, Ion Initial Volume, Dilution Factor, Observed Concentration, Reported Concentration, % Recovery.

7.13 Sample preparation information is entered manually into the Katahdin Information Management System (KIMS). Instrument data files are then imported electronically into KIMS for calculation and reporting of sample results and quality control data. Refer to the current revision of SOP CA-762 ("W et Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

7.14 A batch sheet is generated (Figure 4). Raw data and batch sheets are reviewed for completeness and accuracy by the Department Manager or other qualified designee.

7.15 Printouts of instrument calibrations and sample data are filed in the lab for approximately 3 months for reference by analysts. Prior calibrations are archived and all are available in the TurboChrom database.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Table 2 is a summary of the QC criteria for work following DoD QSM version 4.2. Table 3 is a summary of the QC criteria for work following DoD QSM version 5.0.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Initial Instrument Calibration – Instrument calibration, which is generated using a blank the five standards listed in Section 5.6, is performed at least once every six months or whenever there is a significant change in instrument operating conditions or hardware. One of the calibration standards must be at or below the Practical

Quantitation Limit for each analyte. The curve fit is accomplished using least squares linear regression, and the correlation coefficient for the curve must be at least 0.995. Sample results that exceed the calibration range of the instrument may not be reported; the sample must be diluted and reanalyzed until the measured concentration of the analyte is within the calibration range. Because calibration linearity is established each time the instrument is calibrated (at least every six months) and because sample results that fall outside the calibration range may not be reported, a separate linear calibration range study is not performed.

8.2 Lower Limit of Quantitation - The laboratory should establish the LLOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard prepared at the LLOQ concentration levels or use of the LLOQs as the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LLOQ recoveries must be within 50% of the true values to verify the data reporting limit.

8.3 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) – Ongoing calibration accuracy is verified by analyzing a CCV standard (a mid-range check standard) and a CCB at the beginning of each run, after every ten samples, and at the end of each run. The recovery of each CCV must be within 90% - 110% of the true value for each analyte. The measured concentration of each analyte in the CCB must be below the Practical Quantitation Limit for the analyte. If a CCV or CCB fails, the analysis must be stopped, the problem corrected, and the previous ten samples must be reanalyzed, with the following exception. If one or both CCVs bracketing a sample result are biased high and the sample concentration is <PQL, the sample result may be reported. CCVs or CCBs that are biased high may be indicative of carryover or contamination in the system by high concentration samples.

NOTE: High bias for chloride may be indicative of chloride contamination in the injector. If the run is attended and chloride is an analyte of interest, halt the run and take corrective action if bias is observed.

8.4 Laboratory Control Sample (LCS) / Initial Calibration Verification (ICV) – One LCS/ICV, prepared from a separate standard source from the Initial Instrument Calibration, must be analyzed with each batch of 20 or fewer samples. LCS/ICV recovery acceptance limits are 90% - 110% for EPA Method 300.0 and 80% - 120% for SW846 Method 9056.

For DoD QSM 5.0, use QC acceptance criteria specified by DoD, if available). Otherwise use in-house control limits.

8.5 Method Blank (MB) – A method blank consisting of reagent water that filtered in the same fashion as the associated samples must be analyted with each batch of 20 or fewer samples. The measured concentration of each analyte in the MB must be less

than the PQL (for DoD QSM, no analyte may be detected in the MB at a concentration greater than ½ PQL or greater than 1/10 the amount measured in any sample). In the instance where there is a value in excess of the CCB, the filter source should be suspected as contributing contamination. Repeat the analysis with additional cartridges.

8.6 Matrix Spike (MS) Sample – Matrix spike samples must be prepared and analyzed at a frequency of 10% (one sample in 10) for EPA Method 300.0 or 5% (one sample in 20) for SW846 Method 9056A. Matrix spikes is prepared by adding 0.05 mL of Primary Matrix Spike Mixed Standard (Section 5.9) to 1.0 mL of the filtered sample prior to loading. If the concentration of the spike is less than 25% of the native sample concentration the matrix spike recovery should not be calculated. The matrix spike recovery acceptance limits are 90% - 110% for EPA Method 300.0 and 80% - 120% for SW846 Method 9056A. If the recovery of any analyte falls outside the criteria range and the LCS and CCVs are within criteria, the poor recovery should be attributed to sample matrix.

For DoD QSM 5.0, use QC acceptance criteria specified by DoD, if available). Otherwise use in-house control limits.

8.7 Matrix Spike Duplicate (MSD) – Prepared at a frequency of one per 20 samples. Acceptance limits are 80% - 120% recovery and ≤15% RPD.

8.8 Laboratory Duplicate (Dup) - One duplicate sample must be analyzed with each batch of 20 or fewer samples (one per 10 samples for DoD QSM). Analytes with measured values ≥ 5 times the PQL should achieve duplicate/MSD sample precision of ≤ 20% RPD (≤10% for DoD QSM).

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses

utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Inorganic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of the applicable methods for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Methods for the Determination of Inorganic Substances in Environmental Samples", EPA - 600/R - 93 - 100, August 1993.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, Third Edition, Final Update IV, February 2007, Method 9056A.

Katahdin SOP CA-101, Equipment Maintenance and Toubleshooting, current revision.

Katahdin SOP CA-762, Wet Chemistry Data Entry and Review Using Katahdin Information Management System (KIMS)

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Current Version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

WISP717plus Autosampler Operator Manual

Waters 510 HPLC Pump Manual

Dionex 200i Manual

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

LIST OF TABLES AND FIGURES

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Anions by Ion Chromato-graphy / EPA Method 300.0 and SW846 Method 9056A	Retention time (RT) window width calculated for each analyte	After method setup and after major maintenance (e.g. column change)	RT width is ± times standard deviation for each analyte over a 24-hour period.	N.A.
	Initial Instrument Calibration (ICAL): Blank + 5 standards, lowest standard at or below PQL	Every 6 months or with each change in instrument operating conditions or equipment	1) Correlation coefficient ≥ 0.995 2) Recovery of lowest standard within 50%-150%	Correct problem and recalibrate
	Retention time window established for each analyte	Once per ICAL or at the beginning of each day of use	Position shall be set using the midpoint standard of the ICAL when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	N.A.
	Method blank	One per prep/analysis batch of 20 or fewer samples	No analyte detected ≥PQL	(1) Investigate source of contamination (2) Report all sample results <PQL. (3) Report sample results >10X the blank result and flag results with a “B”. (4) Reanalyze all other samples associated with the failing blank.
	LCS/ICV	One per prep/analysis batch of 20 or fewer samples, prepared from a separate source than calibration standard	90%-110% Recovery	(1) If the ICV/LCS fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.
	CCV	At beginning of run, after every 10 samples, and at end of run	(1) 90%-110% recovery. (2) All analytes within estabilished RT windows.	(1) If the CCV fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze samples back to last acceptable CCV recovery.

TABLE 1

QC REQUIREMENTS (Continued)

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Anions by Ion Chromato-graphy / EPA Method 300.0 and SW846 Method 9056A	CCB	Immediately following each CCV	No analyte detected ≥PQL	1) Investigate source of contamination (2) Report all sample results <PQL. (3) Report sample results >10X the blank result and flag results with a "B". (4) Reanalyze all other samples associated with the failing CCB.
	Matrix Spike	One for every set of 10 samples (EPA 300.0) or one for every set of 20 samples (SW846 9056A)	90%-110% recovery (EPA 300.0) 80%-120% recovery (SW846 9056A)	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, flag result and narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.
	Matrix Spike Duplicate	One per 20 samples	(1) 90%-110% recovery (EPA 300.0) 80%-120% recovery (SW846 9056A) (2) RPD ≤15%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still out, report original result with flagging and narration.
	Sample Duplicate	One per 20 samples	RPD ≤20%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still out, report original result with flagging and narration.
	Demonstration of analyst proficiency; accuracy and precision	One time initially by each analyst performing the method and annually thereafter.	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personnel training file
	MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise use method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	(Refer to current revision of SOP QA-806)				
LOQ establishment and verification	(Refer to current revision of SOP QA-806)				
Retention time (RT) window width calculated for each analyte	After method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT over a 24-hour period.	NA.	NA.	
Initial calibration (ICAL) for all analytes (minimum three standards and one calibration blank)	ICAL prior to sample analysis.	r ≥ 0.995.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Initial calibration verification (ICV) (second source)	Once after each ICAL, prior to beginning a sample run.	All analytes within ± 10% of true value and retention times within appropriate windows.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte	Once per multipoint calibration.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Midrange continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All project analytes within established retention time windows. Within ± 10% of true value.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results (see Box D-1).	Correct problem, then see criteria in Box D-1. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported	One per preparatory batch.	Laboratory in-house limits not to exceed ± 20%. Control limits may be not greater than ± 3 times the standard deviation of the mean LCS recovery. See Box D-3.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use laboratory in-house LCS limits (not to exceed ± 20%).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use laboratory in-house LCS limits (not to exceed ± 20%). RPD ≤ 15% (between MS and MSD).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample duplicate (replicate)	The data shall be evaluated to determine the source of difference.	Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.	Correct problem and reanalyze sample and duplicate.	One per every 10 samples.	%D ≤ 10% (between sample and sample duplicate).
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TABLE 3

DoD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes	ICAL prior to sample analysis.	r2 ≥ 0.99.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Minimum 3 standards and a calibration blank. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per multipoint calibration.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Established for each analyte.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT over a 24-hour period.	NA.	NA.	Calculated for each analyte.
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Freshly prepared ICV. No samples shall be analyzed until calibration has been verified.
Continuing Calibration Verification (CCV)	Before sample analysis; after every 10 field samples; and at the end of the analysis sequence.	All reported analytes within established retention time windows. All reported analytes within ± 10% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method Blank (MB)	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TABLE 3

DoD QSM 5.0 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for all reported analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Follow project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all reported analytes. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, (i.e., matrix effect or analytical error.)
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 15% (between MS and MSD or sample and MD).	Follow project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all reported analytes. The data shall be evaluated to determine the source of difference.

TABLE 4

EXAMPLE ANALYTICAL SEQUENCE WITH ACCEPTANCE CRITERIA

Sample Number	Instrument Runlog	Acceptance Limit
1	CCV	90%-110%
2	CCB	< PQL
3	ICV / LCS	90 -110% (300.0) 80%-120% (9056A)
4	Sample 1	
5	Sample 2	
6	Sample 3	
7	Sample 4	
8	Sample 5	
9	Sample 6	
10	Sample 7	
11	Sample 8	
12	Sample 9 -Duplicate	20% RPD (10% for DoD)
13	Sample 10 - Matrix Spike	90 -110% (300.0) 80%-120% (9056A)
14	CCV	90%-110%
15	CCB	< PQL
16	Sample 11	
17	Sample 12	
18	Sample 13	
19	Sample 14	
20	Sample 15	
21	Sample 16	
22	Sample 17	
23	Sample 18	
24	Sample 19	
25	Sample 20 - Matrix Spike	90 -110% (300.0) 80%-120% (9056A)
26	CCV	90%-110%
27	CCB	< PQL

TABLE 5

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-742-09	Method 300.0/9056, current revisions
Calibration	1) Calibration consists of blank + 5 standards 2) Linearity verified by performing calibration at least every 6 months, and diluting all samples exceeding calibration range	1) Calibration consists of blank + at least 3 standards 2) Linear range verification required every 6 months (Method 300.0)
QC – Method Blank	Acceptance limit < PQL	Acceptance limit <MDL (Method 300.0) Acceptance limit <10% of LLOQ or regulatory limit or lowest sample (Method 9056A)
QC – LCS/ICV	Combined LCS and ICV with tighter acceptance limits (90%-110% recovery)	LCS acceptance limits 80%-120%, ICV acceptance limits 90%-110% (Method 9056A)
QC – Duplicate / Matrix Spike Duplicate	RPD acceptance limit ≤20%.	RPD acceptance limit ≤15% for samples at or above midrange, ≤50% for samples near LLOQ (Method 9056A)

FIGURE 1

IC SYSTEM CONFIGURATION

ICS-2000 Ion Chromatography System

A typical IC analysis consists of six stages (see Figure 1-1).

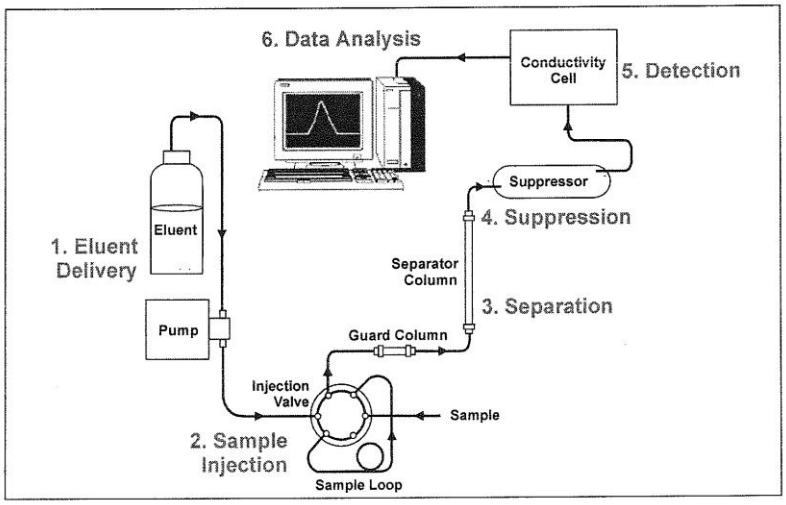

Figure 1-1. Ion Analysis Process

1. **Eluent Delivery**
   - Eluent, a liquid that helps to separate the sample ions, carries the sample through the ion chromatography system. The ICS-2000 includes an eluent generator, which generates eluent online from deionized water.
   - When the ICS-2000 is controlled from the front panel, only isocratic eluent delivery is possible. This means that the eluent composition and concentration remain constant throughout the run. Gradient delivery (a change in concentration over time) is possible when the ICS-2000 is controlled by Chromeleon® Chromatography Management System (the data collection system for the ICS-2000).
2. **Sample Injection**
   - The liquid sample is loaded into a sample loop either manually or automatically (if an automated sampler is installed). When triggered, the ICS-2000 injects the sample into the eluent stream.

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

FIGURE 2

EXAMPLE OF IC MAINTENANCE LOG PAGE

KATAHDIN ANALYTICAL SERVICES, INC.
WATERS IC (ION CHROMATOGRAPHY) - SERIAL NO. 001457
MAINTENANCE LOGBOOK

DAILY INSTRUMENT PERFORMANCE

	Date / Initials	Date / Initials	Date / Initials	Date / Initials	Date / Initials	Date / Initials
Record Line Pressure						
	√	√	√	√	√	√
Detector B Base Range - 50						
Detector B Sensitivity - 0.05						
WISP Injector Volume - 20 uL						
WISP Runtime - 10 Min.						
Instrument run w/ no samples						

ROUTINE MAINTENANCE (As Needed)

Task	Date	Initials	Comments
Clean surfaces of system unit as needed.			
Check regenerate pump tubing & replace as needed.			
Clean or regenerate column as needed.			
Change analytical column as needed.			
Change guard column as needed.			
Change suppressor as needed.			

NON ROUTINE MAINTENANCE (Date and Initial each entry)

- 5/5/12 cleaned anion guard and analytical columns w/ oxalic acid to sharpen PO_4 peak.
- 5/09/12 PO_4 RT - 8.59

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QAWL608

0000014

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

FIGURE 3

EXAMPLE OF ANIONS BY IC RUNLOG PAGE

Katahdin Analytical Services, Inc **ION CHROMATOGRAPHY RUNLOG** Dionex ICS-2000 (Instrument IC-2)

Analysis Date: 06/26/12	**Analytical Column S/N:** 003709	**Calibration Date:** 06/26/12
Analysis Sequence: 06262A	**Guard Column S/N:** 003612	**Calibration Sequence:** 06262A
Analyst: GFB	**Suppressor S/N:** 120329008	☐ If box at left is checked, continued from previous page. Refer to previous page for header information.

Reporting / Reanalysis Codes: ✓ Report without manipulation; S Report, peak automatically reintegrated with SmartPeak; M Report, peak manually integrated; A Report, peak manually assigned; R Do not report, reanalyze sample

Method Codes: E EPA 300.0; SW SW846 9056A

Katahdin Sample Number	Dilution Factor	Method Code	Report or Reanalyze (enter appropriate code): F	Cl	NO_2	SO_4	Br	NO_3	PO_4	Comments
Blank	1.0	E								
Blank										
Cal Std 1				✓	✓	✓	✓	✓	✓	
2				↓	↓	↓	↓	↓	↓	
3										
4										
5										
6										
CCV										
CCB										
Method Blank										
LCS				↓	↓	↓	↓	↓	↓	
3F38806-1				R	✓	✓	✓	✓	✓	Dilute + rerun Cl
-1 Dup					✓	✓	✓	✓	✓	
-1 MS	↓				✓	✓	✓	✓	✓	
-1	10.			✓						
-1	100.									
LOQ Soil NO2	1.0				✓					NO_2 spiked at 0.1 mg/L (10 mg/kg)
CCV	↓			✓	✓	✓	✓	✓	✓	
CCB	↓	↓		✓	✓	✓	✓	✓	✓	
										06/27/12

Reviewed by: _____ Review Date: _____

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

FIGURE 4

EXAMPLE OF IC BATCH SHEET

WET CHEMISTRY BATCH REPORT
May 29 2012, 10:02 am
Batch: WG108595

Parameter: Sulfate
Prep Date: N/A

Date Analyzed: 24-MAY-12
Prep Method: N/A

Analyst Initials: CT
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SF2996-1	SAMP	SW846 9056	1.0000mL	1.0000mL	1	17.7831 ✓	18. mg/L	NA	1	0.064	1.0		
SF2996-2	SAMP	SW846 9056	1.0000mL	1.0000mL	1	2.1434 ✓	2.1 mg/L	NA	1	0.064	1.0		
SF2996-3	SAMP	SW846 9056	1.0000mL	1.0000mL	1	2.0938 ✓	2.1 mg/L	NA	1	0.064	1.0		
SF2996-4	SAMP	SW846 9056	1.0000mL	1.0000mL	1	2.133 ✓	2.1 mg/L	NA	1	0.064	1.0		
SF2996-5	SAMP	SW846 9056	1.0000mL	1.0000mL	1	3.3662 ✓	3.4 mg/L	NA	1	0.064	1.0		
WG108595-1	MBLANK	SW846 9056	1.0000mL	1.0000mL	1	.0314 ✓	U0.50 mg/L	NA	1	0.064	1.0		
WG108595-2	LCS	SW846 9056	1.0000mL	1.0000mL	1	3.703 ✓	3.7 mg/L	NA	1	0.064	1.0		99
WG108595-3	MS	SW846 9056	1.0000mL	1.0000mL	2	21.0206 ✓	21. mg/L	NA	1	0.13	2.0		86
WG108595-4	MSD	SW846 9056	1.0000mL	1.0000mL	2	21.0358 ✓	21. mg/L	NA	1	0.13	2.0	0	87

Comments:

SF2996-1	MS/MSD
WG108595-1	SF2863-6
WG108595-2	SF2863-6
WG108595-3	SF2996-1
WG108595-4	SF2996-1

Entered by: CT Date: 5/29/12 Accepted by: [signature] Date: 05/30/12

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Ryan Oliver

Review Date: 3/4/16

SOP Number: CA-742-10

SOP Title: Anions by Ion Chromatography using EPA method 300.0 and SW-846 9056

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: R Brewer **Date:** 03/23/16

QAO Signature: Leslie Dimond **Date:** 03.25.16

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

Prepared By: Chad Delaney Date: 07.14.04

Approved By:

Department Manager: George Brewer Date: 07/14/04

Operations Manager: Deborah J. Nadeau Date: 7.14.04

QA Officer: Maria Crouch Date: 07.14.04

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Revised Title and text to incorporate analysis for Total Inorganic Carbon (TIC) minor changes throughout Updated Figures	LAD	04/06	04/06
02	Changed SM5310B to SM5310C throughout. Added reference to SW846 9060 throughout. Updated Figure 6. Updated Table 2 to include that we do not subtract the blank from the sample.	LAD	03/07	03/07
03	Added LCS/ICV, CCV, and CCB definitions. Added calibration criteria. Added quadruplicate criteria for SW9060. Added repeat injection for SM 5310B. Added wording regarding SC criteria to Sect. 8. Changed CCV criteria to 90>x<110% LAD	LAD	06/08	06/08
04	Updated reference from 9060 to 9060A. Sect. 7.9 - Changed C "npoc AQ. met for TOC/DOC to C "double inject. met for TOC/DOC.	LAD	03/09	03/09
05	Added EHSM, QA-806 ~~subsampling~~ DRY 8.10.9 DOD, NELAC & CA-161 references.	DN	08/09	08/09

TITLE: ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Removed figures 1 and 2. Updated figures 2-4 (formally 4-4). Updated references.	LAD	06/10	06/10
07	Sect. 5 - Updated calibration levels. LCS and MS prep. Sect. 7 - Added regeneration of TC and IC catalysts. Added stand-by mode and shutting instrument down instructions. Added data archived. Sect. 9 - Added MQL LOD and LOQ info. Sect. 10 - Added & updated references.	LAD	05/12	05/12
08	Sect. 10 - Updated and added References. Updated Fig. 1 & 4	LAD	07/14	09/14

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-763-08** titled **Analysis of TOC, DOC, and TIC in Aqueous Samples using the Shimadzu Carbon Analyzer: EPA Method 415.1, SW846 9060 and SM5310B**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-763-08** titled **Analysis of TOC, DOC, and TIC in Aqueous Samples using the Shimadzu Carbon Analyzer: EPA Method 415.1, SW846 9060 and SM5310B**.

Recipient: _____ Date: _____

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, Inc. technical personnel for analyzing total organic carbon (TOC), dissolved organic carbon (DOC), and total inorganic carbon (TIC) in aqueous samples using the Shimadzu Carbon Analyzer in accordance with EPA method 415.1, SW846 9060A and SM 5310B.

This procedure applies to drinking and surface waters, domestic and industrial wastewater. The Practical Quantitation Limit (PQL) is 1 mg/L.

1.1 Definitions

Total Organic Carbon (TOC) – Carbon that is bound with hydrogen or oxygen in organic compounds, analyzed from an unfiltered sample.

Dissolved Organic Carbon (DOC) - Carbon that is bound with hydrogen or oxygen in organic compounds, analyzed from a filtered sample.

Total Inorganic Carbon (TIC) – Carbon contained in carbonates, hydrogen carbonates, or dissolved carbon dioxide.

Method Blank - A laboratory reagent grade water sample that is carried through the entire analytical procedure in the same manner as a sample.

LCS/ICV - Laboratory Control Sample/ Initial Calibration Verification. One LCS/ICV per batch is prepared from a separate source from the CCV and calibration curve standards. LCS/ICV verifies the calibration curve.

CCV - Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is run every ten samples.

CCB - Continuing Calibration Blank. The CCB is laboratory reagent grade water with no reagents added. One CCB is run every ten samples.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the preparation and analysis of samples for TOC, DOC, and TIC using EPA Method 415.1, SW846 9060 and SM5310 B. Each analyst must demonstrate his/her ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability.”

It is the responsibility of all Katahdin technical personnel involved in the determination of TOC and DOC to read and understand this SOP, to adhere to the

procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health & Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Waste generated during the preparation and analysis of samples must be discarded appropriately. Place all analyzed samples, standards, and rinsings in a Satellite Waste “A” Acid for proper disposal in main waste area “A”.

Other wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Analytical Environmental Health & Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

2.0 SUMMARY OF METHOD

Organic carbon analysis – Sample is drawn into a syringe in the instrument, and hydrochloric acid is then drawn into the syringe to acidify the sample to pH 2 to 3. Oxygen is then bubbled through the acidified sample in the syringe to drive off the inorganic carbon component. The sparged sample is then introduced into the total carbon combustion tube, where it is heated to 680^{o} C in the presence of an oxidation catalyst. The organic carbon remaining in the sample after sparging is converted to carbon dioxide during combustion. Carrier gas sweeps the sample combustion gases through an electronic dehumidifier, where they are cooled and dehydrated, and then through a halogen scrubber to remove chlorine and other halogens. Finally, the carrier gas delivers the combustion gases to the cell of a non-dispersive infrared (NDIR) gas analyzer, where carbon dioxide is detected and quantitated. The amount of carbon dioxide in a sample is directly proportional to the concentration of organic carbon (TOC or DOC) in the sample. Strictly speaking, this method measures non-purgeable organic carbon (NPOC), because purgeable (volatile) organic compounds are lost during sparging of the sample. However, the amount of purgeable organic compounds in natural waters is small, and NPOC is nearly equivalent to total organic carbon. When necessary, total organic carbon can be indirectly determined by measuring total carbon on an unsparged sample, measuring inorganic carbon on a separate aliquot of the sample, and calculating TOC by difference.

Inorganic carbon analysis – Inorganic carbon (IC) consists of the carbon contained in carbonates and in carbon dioxide dissolved in water. IC is measured by injecting an aliquot of unpreserved sample into the instrument's IC reaction vessel, where it is acidified with hydrochloric acid and inorganic carbon is converted to carbon dioxide. The carbon dioxide is then sparged from solution with oxygen and carried to the NDIR detector, where it is detected and quantitated.

3.0 INTERFERENCES

High salt samples can affect the oxidation rate, leading to low recoveries. Interference begins at ~0.1% total dissolved solids and becomes severe at > 0.35%.

4.0 APPARATUS AND MATERIALS

4.1 Shimadzu Carbon Analyzer Model TOC-V

4.2 Shimadzu carousel autosampler Model ASI-V

4.3 Glass 40 mL VOA Vials

4.4 Class A volumetric flasks (100 mL and 200 mL)

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

4.5 Adjustable pipets (0.1, 1.0 and 5.0 mL)

4.6 Oxygen gas

5.0 REAGENTS

NOTE: All standards and reagents should be prepared <u>daily</u> except where noted. Record all standard and reagent preparation in the appropriate logbook.

5.1 Phosphoric acid (H_3PO_4), concentrated, reagent grade.

5.2 Phosphoric acid (H_3PO_4), 1:1 – Slowly add 100 mL concentrated H_3PO_4 to 100 mL and mix. Prepare fresh every six months.

5.3 Hydrochloric acid (HCl), concentrated, reagent grade.

5.4 Hydrochloric acid (HCl), 2M – Slowly add 42.5 mL concentrated HCl to 200 mL of reagent water and bring to a final volume of 250 mL. Prepare fresh every six months.

5.5 Potassium hydrogen phthalate ($HOCOC_6H_4COOK$), primary standard grade. Two different lots of potassium hydrogen phthalate are required – one is used to prepare organic carbon calibration standards, and the other is used to prepare a laboratory control sample (second source).

5.6 Sodium carbonate (Na_2CO_3), 1 N – Purchased certified standard solution, inorganic carbon true value = 6000 mg/L. This solution is used to prepare the inorganic carbon stock standard and the alkalinity check standard.

5.7 Sodium carbonate (Na_2CO_3), anhydrous – ACS reagent grade. Store in a desiccator. This reagent is used to prepare the inorganic carbon laboratory control sample.

<u>Organic Carbon (OC) Standards</u>

5.8 OC Stock Standard (used in preparing OC calibration standards and CCV): Prepare a 2000 mg/L standard by dissolving 0.4250g of potassium hydrogen phthalate in 100 mL of laboratory reagent grade water. Add 0.10 mL of concentrated phosphoric acid. Stored in an amber bottle in the refrigerator, this solution may be used for three months.

5.9 OC Calibration Standards – Prepare a series of five calibration standards by diluting the OC Stock Standard with reagent water in accordance with the following table:

Calibration Std. Conc. (mg/L OC)	mL of OC Stock Std. Added	Final Volume (mL)
1.0	0.10	200
5.0	0.50	200
20.0	2.0	200
50.0	5.0	200
200.0	20.0	200

5.10 OC CCV Standard - Prepare a 100 mg/L standard for the CCV by diluting 25 mL of the OC Stock Standard to 500 mL using laboratory reagent grade water. Stored in an amber bottle in the refrigerator, this solution may be used for three months.

5.11 OC Laboratory Control Sample (LCS) – Prepare a 50 mg/L standard from a different source from the calibration standards, as follows. Fill a 1 L volumetric flask half full with laboratory reagent grade water and add one mL of concentrated phosphoric acid as a preservative. Add 0.1062 g of potassium hydrogen phthalate and bring to a final volume of 1 L with laboratory reagent grade water. Stored in an amber bottle in the refrigerator, this solution may be used for three months.

5.12 OC Matrix Spike (MS) sample – OC matrix spike aliquots are prepared by adding 2.0 mL of OC Stock Standard to 38 mL of sample in a VOA vial. The added matrix spike concentration is 100 mg/L.

5.13 Alkalinity Check Sample - Prepare by diluting 2.5 mL of 1.0 N Na_2CO_3 to one liter with laboratory reagent grade water. The inorganic carbon content of this solution is 15 mg/L. This standard is used to monitor for false positives during OC analysis from incomplete removal of inorganic carbon.

Inorganic Carbon (IC) Standards

5.14 IC Stock Standard (used in preparing IC calibration standards and CCV): Prepare a 2000 mg/L standard by diluting 100 mL of 1 N Na_2CO_3 to a final volume of 300 mL in a graduated cylinder. Stored in a glass bottle in the refrigerator, this solution may be used for three months.

5.15 IC Calibration Standards – Prepare a series of five calibration standards by diluting the OC Stock Standard with reagent water in accordance with the following table:

Calibration Std. Conc. (mg/L IC)	mL of IC Stock Std. Added	Final Volume (mL)
1.0	0.10	200
20.0	2.0	200
100.0	10.0	200
200.0	20.0	200

5.16 IC CCV Standard - Prepare a 100 mg/L standard for the CCV by diluting 25 mL of the OC Stock Standard to 500 mL using laboratory reagent grade water. Stored in a glass bottle in the refrigerator, this solution may be used for three months.

5.17 IC Laboratory Control Sample (LCS) – Prepare a 100 mg/L standard from a different source from the calibration standards, as follows. Fill a 250 mL volumetric flask half full with laboratory reagent grade water and add 0.2208 g of anhydrous sodium carbonate. Mix the solution until the sodium carbonate has dissolved, and bring to a final volume of 250 mL with laboratory reagent grade water. Stored in a glass bottle in the refrigerator, this solution may be used for three months. Prepare for analysis by adding 20 mL of the 100 mg/L standard to 20 mL of DI Water in a VOA vial. The final concentration is 50 mg/L.

5.18 IC Matrix Spike (MS) sample – IC matrix spike aliquots are prepared by adding 1.0 mL of IC Stock Standard to 39 mL of sample in a VOA vial. The final matrix spike concentration is 50 mg/L.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Collect samples in glass jars or VOA vials. For organic carbon analysis, preserve with H_2SO_4 at time of collection and store at 4 (±2) °C prior to analysis. For inorganic carbon analysis, do not add acid to the sample, but store at <6 °C, without freezing, prior to analysis. The holding time for TOC, DOC, and IC analysis is 28 days from the time of collection.

If dissolved organic carbon (DOC) is to be determined, the sample must be filtered through a 0.45 micron glass fiber filter prior to preservation with H_2SO_4. If the sample has not been filtered in the field, sample log-in must inform the Inorganic Department Manager that samples need to be filtered upon receipt by the laboratory. Please refer to the current revision of Katahdin SOP SD-902, Sample Receipt and Internal Control, section 7.7.5, for further filtration procedures.

7.0 PROCEDURES

ANALYZER START-UP PROCEDURE

7.1 Turn on the TOC-V module, computer, and oxygen tank.

7.2 Fill rinse water reservoir with laboratory reagent grade water. Verify that there is sufficient room in the satellite waste container to contain the day's waste. Verify that the instrument's humidifier water level is between the "High" and "Low" lines**,** and add laboratory reagent grade water if needed. Also verify that the diution water, hydrochloric acid, and phosphoric acid reservoirs are full.

7.3 Carrier gas flow rate should be set at 150 mL/min. The oxygen pressure should read 200 kPa on the gauge inside the instrument.

7.4 From the main screen, double-click on the **TOC-Control V** icon.

7.5 Double-click the **Sample Table Editor** icon. Enter the User ID and Password.

7.6 At the menu bar, click on File , then click New. Double-click on the **Sample Run** icon.

7.7 Make certain that the System setting is set for "TOC-Vcph/ASI-V", then click OK.

7.8 Click on the **Connect (yellow lightning bolt)** icon to connect the computer to the instrument. Click "Use Settings on PC". This turns on the furnace inside the instrument, and turns on the gas flows. The furnace must be allowed to heat up for one half hour before beginning analysis.

7.9 When the instrument is ready, the TC o or the IC catalyst needs to be regenerated. At the menu bar select Instrument, scroll down to maintenance and select either TC Regenerate or IC regenerate. Once selected a new window will drop down. Click on Start and allow the procedure to finish. When the regeneration is complete, close the window. The instrument is now ready for sample analysis.

SAMPLE ANALYSIS

7.10 At the menu bar, click on Insert, then click Auto Generate. Double-click on the appropriate method ("doubleinjection.met for TOC/DOC, "INORG CARB AQ.met" for inorganic carbon). Then click Next.

7.11 Enter the number of samples as "60" and the start vial as "5", then click Next.

7.12 At the Calibration Curve screen (Screen 3), click Next. You must verify the calibration curve is current (within three months of analysis) by going into the NPOC Method Menu. If the curve is more than three months old, a new curve must be run.

7.13 Click to put a checkmark in all three boxes (beside "At The Beginning", "After Every Ten Samples", and "At The End of Sample Group") under Quality Control.

7.14 Select (1) by clicking on the number (it will highlight the (1) in blue). Click Add. Then click "CCV". This inserts a CCV in autosampler position (1).

7.15 Repeat 7.13 for autosampler position (2), clicking "Blank", and then for autosampler position (3), clicking "LCS".

7.16 Click Finish.

7.17 You may bypass the vial selection screen at this time by clicking OK.

7.18 An autosampler table now appears on the screen. Fill in sample numbers in the "Sample Name" column. If a manual dilution has been performed on the sample in the VOA vial, enter the dilution factor in the "Dilution" column for each diluted sample.

7.19 Unneeded Quality Control and Sample rows may be deleted by selecting each row and then clicking the **Cut (scissors)** icon.

7.20 Place each sample vial in its appropriate position on the autosampler carousel. Matrix spikes are prepared by adding 2 mL of the OC or IC Stock Standard to a VOA vial and adding 40 mL of sample.

7.21 Place the autosampler carousel on the ASI auto sampler and replace the ASI cover.

7.22 Click the **Start (traffic light)** icon, the enter a data file name and click "Save". Data files are named according to the analyte (TOC or TIC), the matrix (AQ), and the date (MMDDYY format), e.g. "TOC_AQ_031505" for an aqueous TOC run performed on 03/15/05.

7.23 Click "Standby".

7.24 An image of the carousel will appear on the screen, along with a table listing the sample numbers and corresponding autosampler positions. Carefully enter the corrected autosampler positions in accordance with the previously printed list that was used to set up the autosampler carousel.

7.25 Click "OK".

7.26 The instrument will then run samples until it has finished with the programmed autosampler sequence.

7.27 When the sequence has finished, the instrument must be set to standby mode before shutting down completely. On the menu bar select standby. Click on Okay. After 30 minutes, the instrument will shut itself down. After the instrument has shut down, turn off the oxygen and click the "Off" button. Though the instrument is already off, the button stills needs to be pressed.

ANALYTICAL QUALITY CONTROL

7.28 A calibration curve is analyzed as necessary but at least every 3 months. Standards are prepared as described in section 5. Acceptance criteria for the calibration is a 0.9950 correlation coefficient for the full curve including the blank.

Operator discretion in approving or rejecting calibrations is encouraged. The decision and rational must be recorded in the instrument log.

A linear calibration applying a first order equation is used to prepare the curve. The equation is:

$$y = mx + b$$

where: y = Instrument response
m = Slope of the line
x = Concentration of the calibration standard
b = The intercept

Acceptance criterion is a 0.995 correlation coefficient or better.

7.29 An analytical batch consists of 20 or fewer field samples (not counting method blanks, laboratory control samples, duplicates, and matrix spikes).

7.30 The operating range of OC and IC analysis for a 150 uL sample injection is 0-200 mg/L. Samples with measured concentrations greater than 200 mg/L must be diluted and reanalyzed.

7.31 Each analytical sequence must start with analysis of a CCV, followed by analysis of a method blank consisting of laboratory reagent grade water. Analyze an second-source laboratory control sample (LCS) with each analytical batch.

7.32 Each TOC or DOC sample is sparged with oxygen to remove residual inorganic carbon. To check for sparging efficiency, analyze an alkalinity check standard near the beginning of each run.

7.33 Samples analyzed by Standard Methods 5310 B must be injected at least twice and have results within ± 10% of each other. If results are greater than ± 10%, the sample should be reinjected until consecutive measurements are within ± 10% of each other.

7.34 Samples analyzed by method SW846 9060 must be analyzed in quadruplicate

7.35 One matrix spike (MS) should be analyzed for every ten (or fewer) samples.

7.36 One sample in every batch of 20 or fewer samples must be analyzed in duplicate. Method SW9060 requires a sample in every batch of 20 or fewer samples be analyzed in quadruplicate.

DATA REVIEW AND REPORTING

7.37 After completion of analysis, instrument data files are imported electronically into the Katahdin Information Management System (KIMS) for calculation and reporting of sample results and quality control samples. After data processing by KIMS, a batch sheet (Figure 2) listing Katahdin Sample Numbers with associated reported results and associated quality control data is printed out of KIMS for each analytical batch. Refer to the current revision of SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

7.38 Analytical data are stored in the instrument's computer during analysis, and data reports are printed after each analytical run has been completed. The instrument data printout shows the signal versus run time plot that was obtained for each sample, as well as the peak area and measured concentration, the dilution factor, and the analysis date and time. A run data summary table (Figure 1) is also printed out from the instrument, containing all of this information except peak plots in analysis order. Copies of the run data summary tables are bound in the Aqueous Carbon Analysis Run Logbook.

7.39 A Carbon Analysis Run Information Sheet (Figure 3) is completed by the analyst for each analytical run. This form lists the analysis methods, dates, and times, calibration information, and the standard IDs for each standard used in the run.

7.40 The raw data package for each analytical run is assembled by the analyst, and consists of the following information (in the order listed):

- KIMS batch sheets (Figure 2) for each batch contained in the run
- The Carbon Analysis Run Information Sheet (Figure 3)
- The run data summary table (Figure 1)
- The raw data printouts which include peak plots
- Associated calibration data

7.41 Data Archival

All batch sheets, raw data, and supporting documents are scanned after final review and the resulting image files are saved on a Katahdin server for use in data package assembly. Image files of raw data are periodically archived by the laboratory's MIS department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some

cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP), in a program specific Quality Systems Manual (QSM) or in state specific criteria. The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

ANALYTICAL RUN QC SAMPLES

8.2 A Continuing Calibration Verification (CCV) standard is analyzed for each analyte after every ten samples and at the end of the analytical run. The CCV solution is as described in Section 5. Results of the CCVs must fall within 90% to 110% of the expected value. If a CCV fails, sample results that are associated with (bracketed by) the failing CCV may not be reported from the run. The samples must be reanalyzed after the problem is corrected and a passing CCV has been analyzed.

8.3 A Continuing Calibration Blank (CCB) consisting of reagent water is analyzed after each CCV. The absolute values of results of CCBs must be less than the Practical Quantitation Level (PQL) for each analyte. If a CCB fails, sample results that are associated with (bracketed by) the failing CCB may not be reported from the run, with the following exception. If the absolute value of a result for a CCB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. All other samples

must be reanalyzed after the problem is corrected and a passing CCB has been analyzed.

8.4 An Alkalinity Check Sample, prepared as described in Section 5.13, is analyzed in each TOC and DOC run. This sample is used to monitor for false positives during OC analysis from incomplete removal of inorganic carbon. The absolute value of results of the Alkalinity Check Sample must be less than the Practical Quantitation Level (PQL) for each analyte. If the Alkalinity Check Sample fails, associated sample results may not be reported from the run. The samples must be reanalyzed after the problem is corrected and a passing Alkalinity Check has been analyzed.

BATCH QC SAMPLES

8.5 A Method Blank, consisting of reagent water, is analyzed with each batch of twenty or fewer samples. The results of method blanks must be less than the Practical Quantitation Level (PQL) for each analyte. If a method blank fails, results for associated samples may not be reported from the batch, with the following exception. If the result for a method blank is greater than the PQL, associated sample results that are greater than or equal to ten times the measured preparation blank concentration may be reported with "B" notation. Associated sample results that are less than the PQL may be reported with no additional notation.

8.6 A laboratory control sample (LCS), prepared as described in Section 5, is analyzed with each batch of twenty or fewer samples. The LCS recovery must be 80-120% for USEPA Method 415.1 and SW846 9060. The LCS recovery must be 90-110% for SM5310B. If a laboratory control sample fails, results for the associated samples may not be reported from the batch, and the samples must be reanalyzed, with the following exception: if the LCS recovery is greater than the upper acceptance limit, samples with measured concentrations below the PQL may be reported with appropriate narration.

MATRIX QC SAMPLES

8.7 Duplicate samples are analyzed at a minimum frequency of one per batch of 20 or fewer samples. The relative percent difference (RPD) between duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = First sample result
D_2 = Second (duplicate) sample result

A control limit of 20% RPD is applied to duplicate analysis. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.8 Matrix spiked samples are prepared at a minimum frequency of one per 10 samples. The recovery for each element in a spiked sample must fall within 75%-125% for USEPA Method 415.1 and SW846 9060 or 80%-120% for SM 5310B if the result for the unspiked sample is less than four times the amount of spike added. If a recovery fails, the associated sample result must be flagged on the report of analysis. The spike recovery should be calculated as follows:

$$\text{Recovery (\%)} = \frac{S-U}{SA} * 100\%$$

where: S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitaion (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.
MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 415.1, SW846 9060 and SM5310B for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

EPA Method 415.1 “Methods for Chemical Analysis of Water and Wastes”, EPA 600/4-79-020, Revised March, 1983.

Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods”, SW-846, third Edition, Final Update IIIB, November, 2004, Method 9060A.

Standard Methods for the Examination of Water and Wastewater, Method 5310 B, High Temperature Combustion Method, 21st Edition, 2005, approved by Standard Method Committee, 2000.

Standard Methods for the Examination of Water and Wastewater, Method 5310 B, High Temperature Combustion Method, 22st Edition, 2012, approved by Standard Method Committee, 2000, editorial revisions 2011.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.2, 10/25/2010.

Department of Defense (DoD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, March, 2013

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

LIST OF TABLES AND FIGURES

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

TABLE 1

QC REQUIREMENTS

Analytical Method/ Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
EPA 415.1 SW846 9060A & SM 5310B TOC/TIC/ DOC	Initial Calibration (including five standards plus blank)	At a minimum every 3 months or as necessary	Linear Regression Correlation Coefficient ≥0.995	(1) Investigate source of problem (2) Recalibrate
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	90%-110% of true value	Reanalyze all associated samples.
	Continuing Calibration Blank (CCB)	After every CCV	Absolute value <PQL	1) If sample result < PQL, or >10x measured CCB value, report result 2) Else, reanalyze
	Method blank	One per analytical batch of 20 or fewer samples	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
	Laboratory control sample (LCS)	One LCS per analytical batch of 20 or fewer samples	80%-120% for EPA 415.1 and SW846 9060 90%-110% for SM5310B	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep the LCS and reanalyze the remaining samples.
	Matrix spike	One MS per ten samples	75%-125% for EPA 415.1 and SW846 9060 80%-120% for SM5310B	If LCS in criteria and matrix interference suspected, flag data
	Sample Duplicate	One sample duplicate per twenty samples	RPD ≤ 20	If lab QC in criteria and matrix interference suspected, flag data
	Initial demonstration of performance (4 replicate analyses of LCS)	Once per analyst per year	Precision and accuracy within method acceptance limits	Correct problem and repeat initial demonstration of performance
	MDL study and/or LOD and LOQ verifications.	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-763-08	METHODS 415.1, SW846 9060 and SM5310B, current revisions
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	None	
Procedures	None	
QC – Blanks	Blank Subtraction not performed	Method SM5310B requires subtraction of method blank concentration from results for associated samples.
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	None	
QC - MDL	None	

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 1

EXAMPLE OF RUN DATA SUMMARY TABLE

	Sample Name	Dilutio	Result	Comment	Date / Time	Vial
1	CCV	1.000	NPOC:94.82 mg/L		06/30/14 06:23:17	1
2	BLANK	1.000	NPOC:0.2570 mg/		06/30/14 06:35:06	2
3	LCS	1.000	NPOC:48.06 mg/L		06/30/14 06:49:06	4
4	ALK CHECK	1.000	NPOC:0.5910 mg/		06/30/14 07:00:53	5
5	SH4749-1	1.000	NPOC:0.2230 mg/		06/30/14 07:12:33	6
6	SH4749-1 MS	1.000	NPOC:95.35 mg/L		06/30/14 07:26:45	7
7	SH4793-4	1.000	NPOC:0.3079 mg/		06/30/14 07:38:29	8
8	SH4793-4 DUP	1.000	NPOC:0.3111 mg/		06/30/14 07:48:18	8
9	SH4016-31	1.000	NPOC:0.3909 mg/		06/30/14 08:00:08	9
10	SH4016-33	1.000	NPOC:0.2564 mg/		06/30/14 08:11:51	10
11	SH4016-45	1.000	NPOC:0.1410 mg/		06/30/14 08:23:18	11
12	SH4016-49	1.000	NPOC:0.3093 mg/		06/30/14 08:34:58	12
13	SH4662-1	1.000	NPOC:1.038 mg/L		06/30/14 08:47:07	13
14	CCV	1.000	NPOC:91.58 mg/L		06/30/14 09:01:34	1
15	BLANK	1.000	NPOC:0.1580 mg/		06/30/14 09:13:07	2
16	SH4662-2	1.000	NPOC:0.5111 mg/		06/30/14 09:25:07	14
17	SH4662-2 DUP	1.000	NPOC:0.5064 mg/		06/30/14 09:34:58	14
18	SH4662-2 MS	1.000	NPOC:92.09 mg/L		06/30/14 09:49:04	15
19	SH4662-2 MSD	1.000	NPOC:91.04 mg/L		06/30/14 10:01:14	15
20	SH4662-3	1.000	NPOC:1.062 mg/L		06/30/14 10:13:19	16
21	SH4734-8	1.000	NPOC:0.8469 mg/		06/30/14 10:25:25	17
22	CCV	1.000	NPOC:93.17 mg/L		06/30/14 10:39:53	1
23	BLANK	1.000	NPOC:0.1535 mg/		06/30/14 10:51:28	2
24						
25						
26						
27						
28						
29						
30						

07/08/14 09:58:45 AM

1/1

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 2

EXAMPLE OF TOC BATCH SHEET (FROM KIMS)

WET CHEMISTRY BATCH REPORT
Jul 08 2014, 10:41 am
Batch: WG146111

Parameter: Total Organic Carbon
Prep Date: N/A

Date Analyzed: 30-JUN-14
Prep Method: N/A

Analyst Initials: ZS
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SH4749-1	SAMP	SM5310B	20.000mL	20.000mL	1	.223	J0.22 mg/L	NA	1	0.10	1.0		
SH4793-4	SAMP	SM5310B	20.000mL	20.000mL	1	.3079	J0.31 mg/L	NA	1	0.10	1.0		
WG146111-1	MBLANK	SM5310B	20.000mL	20.000mL	1	.257	J0.26 mg/L	NA	1	0.10	1.0		
WG146111-2	LCS	SM5310B	20.000mL	20.000mL	1	48.06	48. mg/L	NA	1	0.10	1.0		96
WG146111-3	MS	SM5310B	20.000mL	20.000mL	1	95.35	95. mg/L	NA	1	.1023	1.0		95
WG146111-4	DUP	SM5310B	20.000mL	20.000mL	1	.3111	U1.0 mg/L	NA	1	.1023	1.0	NC	

Comments:

WG146111-1	SH4749-1
WG146111-2	SH4749-1
WG146111-3	SH4749-1
WG146111-4	SH4793-4

Entered by: ZS Date: 7.8.14 Accepted by: D Hadeen Date: 7.8.14

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 3

EXAMPLE OF CARBON ANALYSIS RUN INFORMATION SHEET

KATAHDIN ANALYTICAL SERVICES, INC.
CARBON ANALYSIS RUN INFORMATION SHEET

INSTR. ID: WC2 (Shimadzu TOC-V_{CPH}) **ANALYST:** ZS **DATE:** 6·30·14

FILE NAME: TOC AQ 063014.t32 **METHOD(S):**

	TOC	DOC	TIC
	☐ EPA 415.1	☐ EPA 415.1	☐ EPA 415.1
	☒ SM5310B	☐ SM5310B	☐ SM5310B
	☒ SW9060-TOC1	☐ _____	☐ _____

CALIBRATION DATE: 6·5·14 **CALIBRATION ANALYST:** ZS

Calibration standards were prepared by performing dilutions of the following standard on the day of calibration:

Calibration Source Standard ID	Prep Date	Expiration Date	Standard Conc. (mg/L)
W12310	6·26·14	9·26·14	2000

Note: Calibration must be performed quarterly or whenever a change in analysis conditions warrant. A copy of the associated calibration data is attached to this run.

STANDARDS USED:

Standard Name	Standard ID	Prep Date	Expiration Date	Standard Conc.
CCV	W12311	6·26·14	9·26·14	100 mg C/L
LCS	W12144	4·14·14	7·14·14	50 mg C/L
MS Stock Std. *	W12310	6·26·14	9·26·14	2000 mg C/L
Alkalinity Check	W12188	4·28·14	9·28·14	15 mg/L (Inorg. C)

* Matrix spikes are prepared by adding 2.0 mL of MS Stock Std. to 38 mL of sample.

Additional Comments and Notes:

SMTOC - WG146111
SW9060-TOC1 - WG146112

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: George Brewer

Review Date: 08/20/2015

SOP Number: CA-763-08

SOP Title: ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060, AND SM 5310B

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: George Brewer **Date:** 08/20/15

QAO Signature: Susie Dimond **Date:** 08.20.15

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

Prepared By: Andrea Colby Date: 6/2002

Approved By:

Group Supervisor: Andrea Colby Date: 6/6/02

Lab Operations Mgr: Jeff C. Burton Date: 6/5/02

QA Officer: Deborah J. Nadeau Date: 6.6.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6 7.7.4 7.10 + 7.20. Complete rewrite of sections 7.11 + 7.12 to comply with new KIMS	DN	6.6.02	6.6.02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. Updated new incoming	DN	05.04	05.04
06	Added procedure + logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added cwatoday procedures for food/micro. Added VOA soil freezer storage.	DN	01.26.04	01.26.04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed Figures B and 10. Updated Table and figures w/ current ones. Added wording to sect. 7.7.5 to clarify how pH measurements are taken.	LAD	02/07	02/07
08	Added summary stating sample acceptance policy. Deleted all references to radiation checks (not performed). Add IR gun usage. Reorganized section 7.0 to prioritize time sensitive tasks. Added wireless thermometer monitoring. Updated SRCR. Other minor changes. Added section concerning locking of coolers. Added more detail to 7.18 on unique container IDs. Added more detail on immediate COCs & a section on retention of samples.	DN	~~05/09~~ 08/09 8.4.09	~~05/09~~ 08/09

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
09	Added new login information for bottle IDS & screen attachment. Added procedures for bar code scanning for internal custody & deleted manual forms. Added new Incoming from KIMS & deleted old. Added form controlled forms to figures.	DM	9.24.10	09/10
10	Sect. 7 - Removed F9 function in printing labels, fixed how the lab ID appears on labels, and fixed how the date needs to be entered. Updated Fig.s 6 and 13.	LAD	08/13	08/13
11	Sect. 7 - Updated WC shirts and rushes from log-book to Google Docs, updated microbiological/ food login process, updated bottle labeling. Updated Table 1 and Figures 1→7 & 12. Added Figure 17 - Sample Acceptance Policy	LAD	05/16	05/16

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-902-11**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-11**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services requires the use of specific receiving, acceptance, identification, storage, and distribution procedures for samples it accepts for analyses. These procedures assure that:

- samples are uniquely identified,
- samples are protected from loss or damage,
- essential sample characteristics are preserved,
- any alteration of samples (e.g., filtration, preservation) is documented,
- the correct samples are analyzed, and
- a record of continuous sample custody and utilization is established.

The purpose of this SOP is to describe the procedures used for the receipt and tracking of samples received by Katahdin Analytical Services (Katahdin).

1.1 Definitions

SDG: Sample Delivery Group – A group of samples to be reported as one data package.

1.2 Responsibilities

It is the responsibility of all Katahdin staff who receive samples or handle samples in the course of analysis to follow the procedures set forth in this SOP, to document their understanding of the procedures in their training files (refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”), and to suggest changes and revisions when appropriate. All technical staff are responsible for monitoring their immediate areas, stopping an activity when a problem is detected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., Department Manager, Operations Manager, QAO). The primary responsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the Operations Manager, QAO or President when warranted.

It is the responsibility of the Operations Manager to oversee adherence to Katahdin QA/QC practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the QAO and President.

It is the responsibility of the Quality Assurance Officer (QAO) to oversee adherence to this SOP, to conduct periodic audits of each laboratory, to track corrective action reports, resolution, and documentation, and to communicate concerns and report findings to the President. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Environmental Health & Safety Manual and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the receipt of samples must be disposed of in accordance with the Katahdin Environmental Health & Safety Manual and SOPs SD-903, “Sample Disposal” and CA-107, “The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and

Standards," current revisions. Expired standards are placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

2.0 SUMMARY OF METHOD

Regulatory, program, and/or method requirements dictate the specifics of sample acceptance. These requirements include, but are not limited to, temperature upon receipt, chemical preservation, container type, sample amount, holding time considerations and complete and accurate documentation of all of these conditions, as well as sample identification. Katahdin's sample acceptance policy is to note any anomalies, discrepancies or non-compliances concerning the receipt of samples. The client is always notified with these issues to direct Katahdin on how and whether to proceed with analysis. All guidance from the client is recorded in the project phone logs and/or on the Sample Receipt Condition Report, which becomes part of the final report. Conditions or analyses performed which do not meet the necessary requirements are narrated or notated as described in the individual analytical SOPs.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Thermometer – Oakton® Non-Contact Infrared Thermometer, or equivalent, capable of reading 0.1°C and digital probe style capable of reading 0.1°C (used for back-up).

4.2 Capillary tubes – 75 mm Hematocrit Tubes, disposable

4.3 Wide range pH test strips, pH 0 to 14 pH, EMD ColorpHast or equivalent.

4.4 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.5 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

7.0 PROCEDURES

PROCEDURES FOR SAMPLE CUSTODIAN

The following procedures include all steps to be completed for satisfactory receipt and acceptance of samples at Katahdin. These steps do not necessarily have to be performed in the exact order as described. Sample deliveries occur constantly throughout the day, so the sample custodian must multi-task and move back and forth between different procedures to accomplish the most critical tasks of checking receipt temperatures and checking for "RUSH" or quick hold time parameters.

7.1 When samples (except for non-environmental food samples) are dropped off, by either a delivery service (i.e. FEDEX or UPS) or by the client, the Chain-of-Custody (COC) should be signed immediately. The client (who is delivering or that has shipped samples with a delivery service) shall sign (at the lab upon delivery or prior to shipment of samples) that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figures 1-3 for a Katahdin standard COC, a Katahdin Homeowner COC, and a Katahdin Food/Microbiology COC).

7.2 Cut custody seals and open all coolers. Remove the packets containing the client Chains-of-Custody (COCs).

7.3 Using the COCs, enter the date and time of sample receipt and the client name into the next available work order/login number in the sample receipt logbook (Figure 5). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number. Record the assigned work order numbers in the appropriate space on the client COCs. Complete the log-in entry date and time once samples are logged in as described below.

7.4 Inventory the COCs for any "rush turn around" samples or "short hold-time" analyses. Notify the appropriate department Managers/Supervisors of these analyses.

7.4.1 Short hold-time analyses need to be entered into the "Wet Chemistry Shorts Spreadsheet" (Figure 6) on the company Google Docs system. Be sure to list the client, number of samples and date and time of the earliest sample.

7.4.2 GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion.

7.4.3 Notify all applicable personnel of samples with limited hold-time remaining or rush turn around samples. Appropriate supervisors and PMs must be emailed when a client has requested rush results. The email should include the work order number, the client, the matrix, number of samples, analysis requested and the turnaround time. Samples for microbiology lab should be brought to them immediately.

7.4.4 Parameters that we routinely analyze which have short analytical hold times are:

Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	TBOD	Free CO_2
Sulfite	ENCORE soil samples	Settleable Solids
Odor	Residual Chlorine	CBOD

7.5 Inspect the condition of custody seals, cooler, ice condition and samples received. Note any non-intact conditions on the Sample Receipt Condition Report (SRCR - Figure 7). Notify the Katahdin project manager (PM) of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, close and seal the packing container with all the samples inside and move to a hood in the organic extractions area or to the smaller hood in the login area if space permits. One of the three Katahdin Emergency Response Coordinators or the Katahdin Environmental Health & Safety Manager must be notified. Disposition of the broken and other possibly contaminated samples will be determined on a case-by-case basis in accordance with the laboratory's handling procedures for hazardous waste as outlined in the Katahdin Environmental Health & Safety Manual. Generally, when a sample has broken and has mixed with any ice in the cooler, that liquid will be poured off into 2 liter plastic containers and labeled as "do not use". These containers will be disposed of as soon as the disposition of the appropriate samples has been determined through analysis.

7.6 If there is no breakage of a potentially hazardous sample:

Check cooler temperatures using the IR thermometer assigned to the sample receipt area. If a cooler temperature blank is present, aim the IR gun at the temperature blank; otherwise aim the IR gun at any sample in the cooler if no temperature blank is present. Be sure that the IR gun is within 6 inches of the bottle and not aimed at a label on the bottle. Press the trigger on the handle and be sure the red dot is visible on the bottle surface. The IR gun has been set to read in degrees celcius. If checking the temperature of a plastic bottle, set the emissivity at 0.90. If checking the temperature of a glass bottle (either amber or clear), set the emissivity at 0.85. Refer to Figure 8 for manufacturer’s instructions on changing the emissivity. Record the

temperature on the Sample Receipt Condition Report. Receipt temperatures should be <6 °C, without freezing. Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

Note: Samples received for metals analysis only do not have to meet any temperature receipt requirements.

Note: A probe type thermometer is retained as back-up in case there is a problem with the IR thermometer.

7.7 Note the condition of the ice or ice packs. If the ice has melted and the temperature is out of acceptance criteria, note this on the SRCR. For samples that are hand delivered to the laboratory immediately after collection (i.e. sample collection times are <6 hours old), the temperature blank and/or cooler temperature will most likely not meet the acceptance criteria. The samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Note this on the SRCR. If samples (that were just collected) have not arrived on ice, note this on the SRCR, and start the cooling process as soon as possible after arrival at the laboratory.

Note: All clients must be notified when samples are received that do not meet the appropriate temperature requirements. In these cases, certain regulatory requirements may not be met and may invalidate certain data.

7.11 Notify the PM immediately if there are any discrepancies or problems with sample receipt. The PM will contact the client for information and resolution as necessary. All decisions to proceed or not to proceed with analysis associated with samples received that do not meet specified acceptance criteria (i.e. cooler temperature, preservation, container, etc.) must be fully documented on the SRCR. Although this form is included with all client reports, additional narration or flagging of data may be necessary.

7.12 Review any additional paperwork that accompanies the sample(s) submitted for analysis along with laboratory-generated information. This includes shipping forms, letters, chain-of-custody forms, sample labels, Incoming Sample Reports (generated from KIMS), quotes, memos, etc. These forms may provide details on specific client requests. The Incoming will provide information on specifics for log-in. Refer to Figure 11 for an example.

7.13 Resolve any questions or concerns raised by steps 7.1-7.14 by consulting the correspondence files or client services personnel or communicating directly with the client. Note in the notes section of the SRCR any deviations from normal sample handling or analytical procedures (e.g., client requests analysis although hold-time expired).

7.14 Samples requiring microbiological and/or food analyses are stored in the F/M laboratory walkin. For environmental tests, samples are logged in by the sample receipt department and a copy of the chain of custody is brought with the samples. For non-environmental microbiological tests, a workorder number is assigned by sample receipt but the samples are not logged in. The workorder number, the chain of custody and a copy of the chain of custody are delivered with the samples. The samples are then logged in by the F/M staff. Sample that require both environmental and non-environmental microbiological analyses are usually processed the same as non-environmental samples

7.15 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 12) is generated for the samples received:

7.15.1 Log onto KIMS by entering employee ID under “Username”, employee specific password under “Password” and KIMS under “Database”.

7.15.2 Once logged onto KIMS select “Sample Management” and then “Login”.

7.15.3 Select “New” and the next available Login ID number will automatically be entered. Select “OK” and the Sample Definition screen will open.

Note: If a Work Order number has already been opened, select “change” and type in the appropriate number to access the information.

7.15.4 In the Sample Definition Screen, enter the following information.

Top Section of Screen:

Client ID - Enter client sample description.

ReceiveDate - Enter in date that samples were received in the lab in the format Day-Month-Year (ex. August 23, 2013 is 23-AUG-13).

CollectDate - Enter in date that samples were collected in the format Day-Month-YearTime (ex. 8:30am August 23, 2013 is 23-AUG-13).

TAT - Enter TAT for hardcopy report.

DueDate - Due date will automatically be calculated based on calendar days.

VerbalDate - Manually type in verbal due date.

QuoteRef - Enter quote number if applicable.

Project - Enter project number if applicable.

Account - Enter client specific account number.

Account Name - Account name will automatically be entered.

Collected By - Enter name/initials of sampler listed on COC. If unknown, enter “Client”.

Locator - May be used for client ID information when requested by the project manager.

Site - Enter project site name.

Description - May be used for food descriptions.

Discount - No entry-not currently used.

Priority - No entry-not currently used.

Fact. - No entry-not currently used.

Expected - No entry-not currently used.

Mailed - Data Management will enter the mailed date of the report or SDG right after the report is mailed.

Comments - Enter MS/MSD, verbal due date and any sample irregularities if applicable. Also may be used for long client IDs when requested by the project manager.

OrderDate - Current date is automatically entered.

Middle Section of Screen:

Highlight the first sample in the top section of the screen and then proceed with entries in the middle section of the screen.

Matrix - Enter sample matrix code where

AQ = Aqueous	SLD = Food Solid
SL = Solid, Soil, Sludge	AR = Air
FP = Free Product	SWAB = Swab
WP = Wipe	SAL = Saline

NOAQ = NonAqueous TIS = Tissue
DW = Drinking Water

Product Code - Enter analysis code per test requested on COC. Log-in personnel should refer to Project Incomings, quotes or past Work Orders to aid in the entry of correct product codes.

Type - Product code type will automatically be entered where
S = Stand alone
P = Parent
C = Children

Fact. - No entry-default is 1.

Price - This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.

Cost - No entry needed.

Lev - No entry needed.

Container Type - Container type will automatically be entered. Please change from the various choices if the automatic entry is not correct. This is especially important for volatiles in soil since there are many types of preservations.

Container Key- Make sure “Container Type” is populated. Determine how many bottles there are for each container type. Assign bottles by entering sequential letters for each bottle. For example, sample 1 has six containers, one for metals which we’ll assign container ID, “A”, two for PCBs which we’ll assign container IDs, “B” and “C”, and three for volatiles which we’ll assign container IDs, “D”, “E”, and “F”. The letters should be typed in all in a row with no commas or spaces in between. If 26 bottles per samplenum are exceeded the next ‘key’ would be, ‘A1’, ‘B1’ etc. If no container IDs are needed (i.e. for food or field) it is okay to leave the container key field blank.
After the Container Keys are entered click ‘SAVE’. This will create the containers section in the bottom

section of the screen. This will also initiate the creation of container labels.

Bottom Section of Screen:

Container # - The container ID numbers will automatically fill in for each analysis from the container key information above.

Container Type - The container types will automatically fill in for each analysis from the container key information above.

Current Location - The current location is automatically entered based on the analysis.

Cooler - Currently not used.

pH - Currently not used.

Temperature - Currently not used.

Seal - Currently not used.

Properly Preserved - Currently not used.

Comments - Comments on individual containers may be entered here, i.e. bubble in VOA vial. Comments regarding problems or breaks with internal custody scanning of bar codes are also automatically entered here.

Select Login Info tab at top of screen and proceed with entry:

Login Info - Parameter Data Screen will open. Enter following information

KAS Proj. Manager- Initials of Katahdin person overseeing the project.

Client PO#- Client purchase order.

Project- Project name.

Cooler Temperature- Temperature blanks or cooler temps.

Delivery Services- Method of delivery to the lab.

QC Level- QC Level of report

SDG ID- Sample Delivery Group ID if applicable.

SDG Status- Begin, Continue or End.

Analysis Instructions- PM will enter special instructions regarding project.

Report Instructions- PM will enter special instructions regarding project.

Regulatory List- Not used.
EDD Format- Specific KAS EDD format.

Select “SAVE” and then “CANCEL”.

Addresses - Select “Addresses” and the Address Links screen will open. The billing address is the default address of the account. Enter the client account code under “Project/Account” and select the report to contact under “Address Type”. Select the appropriate boxes for report, report CC and invoice CC. Select “SAVE” and then “CLOSE”.

Refer to Figure 13 for a screen snapshot of the log-in process in KIMS. Log-in personnel should also refer to the current revision of Katahdin SOP, SD-918, KIMS Work Order Approval & Dispatching, for further hints on log-in.

7.15.5 To print the login report, select “Reports”, “Login” and “Login COC”. Enter login number under “Login Number”. Select “OK”, “Run Report” and then “Print”.

7.16 To print labels, select “Reports”, “Login” and “Labels”. Enter login number under “Login/Prelogin”, select “Background (IDXL) (this is the default)”. Select “OK” and then “Print”. After labels print out select “Cancel”.

Note: As stated in “container key” above, each sample bottle is assigned a unique ID. The job is given a work order number. Each different client sample ID is given a numerical number following the work order number and each sample container with the same client ID is given a container ID using alphabetical letters. This

series of work order, sample number and container ID is transcribed throughout the raw data for traceability purposes.

Example: One job containing one client sample with 3 different containers:

SC9001-1A, SC9001-1B, SC9001-1C

Example: One job containing two client samples with 2 different containers for each:

SC9002-1A, SC9002-1B, SC9002-2A, SC9002-2B

7.17 Print the Label Bottle Reference report (under reports tab) for a cross reference to use during labeling. This report will list the bottle type and products related to each Container ID.

7.18 Remove samples from cooler and place them on the counter. Organize them by site ID, in the order of the chain and then by sample analysis.

7.19 Inventory the samples against the chain of custody (COC). If the COC is incomplete, the sample custodian must inform the appropriate Katahdin project manager (PM). The PM may make changes to correct or complete the COC, but all changes must be initialed and dated. Changes must be noted on the SRCR. Any discrepancies between the samples and the COC must also be noted on the SRCR.

7.20 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, check if samples are in proper containers and received correct pretreatment (e.g., filtration, preservation) for the analyses requested. For aqueous parameters requiring preservation, check pH by inserting a clean capillary tube into the sample and dabbing the tube on wide range pH paper. If the pH is not clearly either less than 2 or greater than 12, the appropriate narrow range pH paper must be used. NOTE: The pH of volatile organic (VOA) samples is checked and recorded by the analyst after completion of analysis and not by sample receipt personnel. The used capillary tube is discarded and a new capillary tube is used for each sample.

Additional preservative is added to samples if the pH is not in the range specified in the Sampling and Preservation Requirements Table. No more than 10% of the original sample volume should be added as preservative. If the client has noted that the sample reacts violently (i.e., foams and bubbles) upon preservation, add no more preservative to the sample. Some clients may wish to be contacted if their samples are found to be improperly preserved. Record all preservation discrepancies on the Sample Receipt Condition Report including the lot number of the preservative added. If additional preservative is added, a sticker with the type of preservative must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation area.

Note: If samples are received unpreserved for 200.7 or 200.8 analysis, the samples must be preserved to pH <2 with nitric acid. Samples must be held for 16 hours after preservation before sample preparation can begin.

7.21 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 9). The samples are filtered by the Metals Group or the Wet Chemistry Group depending on which group requires the filtered samples.

7.21.1 A 500 mL filter flask and filter funnel are acid rinsed three times in a 10% nitric acid bath, then three times with Laboratory Reagent Grade Water.

7.21.2 A vacuum pump is attached.

7.21.3 A 0.45 micron filter is rinsed three times with 5% nitric acid and three times with Laboratory Reagent Grade Water. The rinsate is discarded.

7.21.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid to pH <2.

7.21.5 The bottles are labeled with the work order/login number and other sample information and stored at <6 ° C until the time of digestion.

7.22 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, determine if sufficient volume of sample is present for analysis. Note discrepancies on the SRCR.

7.23 For drinking water samples, enter the appropriate information (work order, date, etc.) into the Measured Turbidity and Preservation of Incoming Samples Logbook. Inform the appropriate analyst of the sample. The turbidity must be measured prior to sample preparation. If the turbidity is <1 NTU, the sample does not have to be digested prior to metals analysis. If the turbidity is >1 NTU, the sample must be digested prior to metals analysis. The sample must be preserved after the turbidity measurement is taken. Record the appropriate information in the logbook (Figure 10).

7.24 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Do not obscure client ID on the bottles. 40 mL vial, 125 ml plastic bottle and 4 oz jar labels will have to be placed vertically on the sample container instead of the standard horizontal placement. Additionally, label for 2 oz jars must be placed on the cover.

7.25 Scan the containers into the appropriate storage locations using the following steps. Note that non-environmental food samples are not scanned and are taken immediately to the food/microbiology lab for storage.

7.25.1 In KIMS, click on “containers”. This can also be done at the walk-in computer or on the “D” instrument computer in the VOA lab, depending on where you are storing samples.

7.25.2 Click on “transfer/update” then “transfer” and select. This will bring you to the screen where you scan your badge. **NOTE: make sure you keep your badge available for this**. Alternatively, at the walk-in computer, click on the check-in/check-out ICON. This will also bring you to the screen where you scan your badge.

7.25.3 Scan the barcode on your badge.

7.25.4 Pick “log-in”.

7.25.5 Pick “check-in”.

7.25.6 Select the location you are checking into, i.e. walk-in, VOA Walkin, etc.

7.25.7 The sample screen will now be open. Scan each sample, so that you hear a beep and the sample pops up on the screen. The program is set so that you can continuously scan each sample without having to click anything on the screen. The samples do not have to be scanned in numerical order.

7.25.8 Hit “done/save”.

7.25.9 Hit “close/cancel”. This will return you to the badge scanning screen.

Note: An internal custody report may currently be printed, per client request, by the MIS department.

7.26 Place samples in their designated storage locations. Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walk-in cooler, which is organized by test type and work order/login number.

Specific storage locations are described below.

7.26.1 Aqueous samples for wet chemistry (except hardness, see 7.19.4 below) - left aisle, both sides, as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples.

7.26.2 Aqueous samples for organic extractions – right aisle, left side, as you enter walk-in cooler.

7.26.3 Non-aqueous samples (all analyses except volatile organics) - to the right and towards the back as you enter walk-in cooler. Non-aqueous samples for volatile organics are stored in “VOA Refrigerator 2” located in the Volatiles Laboratory.

7.26.4 Aqueous samples for metals and/or hardness analyses – right aisle, right side towards the front as you enter walk-in cooler.

7.26.5 Samples (aqueous and solid) for volatile organics analyses (VOA) – All aqueous and soil samples in VOA vials (except those which are preserved with D.I. water) are stored in “VOA walkin” in the Volatiles Laboratory. VOA samples known or suspected to be hazardous (such that cross-contamination of other samples might occur) are placed in a “paint can” and stored in the sample receipt walk-in.

7.26.6 Soil samples for volatile organics analyses (VOA) that are preserved with Laboratory Reagent Grade Water are stored in “VOA Freezer 1” in the volatiles laboratory.

Sample storage coolers are not locked, but internal chain-of-custody is documented through the bar code system with respect to native samples. Internal chain-of-custody for extracts and digestates is documented on hardcopy batch sheets. The laboratory maintains a secure facility with respect to unauthorized personnel, as described in the current revision of Katahdin SOP, AD-004, Laboratory Facility Security and Confidentiality. All sample storage coolers are equipped with locks if specific project or regulatory requirements deem it necessary.

7.27 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. Any project or program specific forms should be included with the paperwork at this time. These forms may include CLP forms or state-specific forms. The project manager then dispatches the work order/login to the individual department worklists. The dispatched work order/login package is then filed in Data Management where the complete package will eventually be compiled.

7.28 The temperature of all sample storage refrigerators and freezers is recorded daily by assigned individuals. Notebooks containing a record of each refrigerator and freezer temperature history are used for this purpose and are maintained by the assigned individuals. Temperatures above or below the acceptance range are to be brought to the attention of a Department Manager, Operations Manager, or Quality Assurance Officer. Such an occurrence and the actions taken to correct it must be noted in the comments column of the temperature recording notebook next to the temperature measurement. (See Figure 14).

Additionally, temperatures of storage units are monitored continuously by wireless thermometers. A temperature is recorded electronically every 10 minutes. The QAO can generate a specified report as needed, including every reading or maximum/minimum temperatures for a given timeframe. These monitoring devices ensure continual compliance seven days per week. The data can be used to check for problems.

PROCEDURES FOR CHEMISTS

7.29 When removing or returning a sample from its storage location, it must be scanned in or out using the bar code on the container.

7.29.1 In KIMS, click on “containers”.

7.29.2 Click on “transfer/update” then “transfer” and select.

7.29.3 This will bring you to the screen where you scan your badge. Alternatively, at the walk-in computer, click on the check-in/check-out ICON. This will also bring you to the screen where you scan your badge.

7.29.4 Scan the barcode on your badge.

7.29.5 Pick the department that you are bringing samples to or from.

7.29.6 Pick “check-in” or “check-out”.

7.29.7 For check-in, select the location you are checking into.

7.29.8 The sample screen will now be open. Scan each sample, so that you hear a beep and the sample pops up on the screen.

7.29.9 Hit “done/save”.

7.29.10 Hit “close/cancel”. This will return you to the badge scanning screen.

7.30 If the samples have not been logged in yet and they need to be pulled in order to analyze short holding time parameters, the analyst taking the sample must use the designated logbook (Immediate Internal COC – Figure 15) to sign the samples out. Many circumstances lead to analysts having to pull samples before they are logged into the KIMS system. It is everyone's responsibility to ensure that all samples can be accounted for at all times. Failure to do so can create confusion and bottle necks for others trying to access the samples. Samples that are pulled before log-in must be returned to the designated bin in the sample receipt area. The Immediate Internal COC Logbook must always be consulted if there is ever a question about internal custody.

7.31 If there is an error (i.e. a sample was checked out, but not checked back, and you are trying to check it out), an error screen will pop up indicating who made the error. Take note of who made the error and click "accept bottle". This will allow you to continue, and a note will automatically be applied to the record. If you notice somebody making a lot of errors, please talk to them or let a manager know.

7.32 For samples that are consumed during analysis or preparation, i.e. extractables – either log the samples out and then rescan your badge and log them back in to "consumed" or remove the labels in the lab (when finished) and stick them to your lab coat and then return to scan them into "consumed".

7.33 If a sample is not consumed by an analysis, return the remaining sample to its assigned storage location and rescan back in using the steps in 7.23.

7.34 After the completion of all analyses, the original "left over" sample containers will remain in sample storage until their final disposal. Samples are held during this period for the purposes of retesting if required by a laboratory corrective action or by a client. Refer to the current revision of Katahdin SOP, SD-903, Sample Disposal, for details on final disposal of samples.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each thermometer used to monitor sample storage or cooler temperatures must be calibrated annually against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for annual calibration and for maintaining the calibration records. All other procedures and documentation listed in this SOP must be followed at all times.

9.0 METHOD PERFORMANCE

Not applicable.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories," U.S. EPA EMSL Office of Research and Development, March 1979.

Code of Federal Regulations 40, Parts 136 and 141.

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 Chapters 1 & 2, USEPA, Third Edition, including Updates I, II, IIA, and IIB, III June, 1997.

Katahdin Analytical Services, Environmental Health & Safety Manual, current revision.

Katahdin QA Manual, current revision

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Current Version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

LIST OF TABLES & FIGURES

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES - AQUEOUS					
Acidity	SM 2310B, 305.1	100 mL	P,G	1,2	14 days
Alkalinity- Titrimetric	SM2320B, 310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1/350.2 SM4500NH3 B&H	100 L	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, SM4500NH3 H	100 mL	P,G	1,3	28 days
Anions (F, Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (calculation from alkalinity)	SM4500-CO2 D				
Biochemical Oxygen Demand-Carbonaceous	SM 5210B, 405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	SM 5210B, 405.1	1 L	P,G	1	48 hours
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	SM4500-Cl E, 325.2	100 mL	P,G	1	28 days
Chlorine, Total Residual	SM4500-Cl G, HACH 8167	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	SM2120B, 110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	SM4500CN G, 335.1	100 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.4	100 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	SM4500-O G, 360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1,12	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F B/C, 340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	SM4500F C, 340.2	200 mL	P only	1	28 days
Free CO2	SM4500-CO2 C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2, SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	SM4500-NO3 F, 353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	SM4500-NO3 F, 353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	SM4500-NO3 F, 353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1,11	28 days
pH (Laboratory)	SM 4500H B 150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	SM4500-P E, 365.2	100 mL	P,G	1	48 hours

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PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	SM 2540C, 160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	SM 2540D, 160.2	1 L	P,G	1	7 days
Solids-Settleable Solids (SS)	SM2540F, 160.5	1 L	P,G	1	48 hours
Solids-Total Solids	SM 2540B, 160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	SM 2540E, 160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	SM2540C/E, 160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance	SM2510B, 120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	ASTM D516-02, 375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	SM4500-S2 F, 376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	SM4500-SO3 B, 377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon	SM 5310B, 415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	SM2130B, 180.1	100 mL	P,G	1	48 hours
Volatile Fatty Acids	SOP CA-776	(2) 40 mL	VOA vial	17	14 days
ELEMENTAL ANALYSES - AQUEOUS					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	16	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES - AQUEOUS					
EDB, DBCP & 1,2,3-TCP	8011 & 504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP EPH	(2) 1000 mL	Amber Glass	1,12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Petroleum Range Organics	FL-PRO	(2) 1000 mL	Amber Glass	1,12	7days/40days

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SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Total Petroleum Hydrocarbons	TX1005	(2) 40 mL	VOA vial	12	14days/14days
Extractable Total Petroleum Hydrocarbons	CT-ETPH	(2) 1000 mL	Amber Glass	1	7days/40days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days
PCB Congeners	8082	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP VPH	(2) 40 mL	VOA vial	1,11	14days
Chloropicrin	8011 Mod.	(2) 40 mL	VOA vial	1,8,9	14 days
HPLC ANALYSES - AQUEOUS					
HPLC-Explosives	8330A/B/ B Mod.	(2) 1000 mL	Amber Glass	1	7days/40days
GC/MS ORGANIC ANALYSES - AQUEOUS					
Acid Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles - Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(~)
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables & (SIM)	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics & (limited SIM)	8260/8260 SIM	(3) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics	624	(3) 40 mL	VOA vial	1,8,9	14 days(~)
MICROBIOLOGICAL ANALYSES - AQUEOUS					
Coliform, Fecal (wastewater)	SM 9222D	100 mL	P,G	1,6	6 hours
Coliform, Fecal (wastewater)	Colilert-18 w/ Quantitray	100 mL	P,G	1,6	6 hours
Coliform, Total (wastewater)	SM 9222B	100 mL	P,G	1,6	6 hours
Coliform, Total (drinking water)	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total (drinking water)	SM9223B, Colitag	100 mL	P,G	1,6	30 hours
E-coli (wastewater)	SM9213D	100 mL	P,G	1,6	6 hours
E-coli (wastewater)	SM9223B Colilert w/ Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B, SIMPlate	100 mL	P,G	1,6	30 hours
GENERAL CHEMICAL ANALYSES – SOLID					
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

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SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Ammonia-Nitrogen-Automated Phenate	350.1/350.2 SM4500NH3 B&H mod.	4 oz	Soil Jar	1	28 days (^)
Anions (F, Cl, Br, NO3, NO2, SO4)	9056	4 oz	Soil Jar	1	48hrs to 28 days (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/9056	4 oz	Soil Jar	1	28days (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	SM4500F B/C, 340.2 mod.	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	9056 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	9056 mod./353.2	4 oz	Soil Jar	1	48 hrs (^)
Nitrite-Automated Diazotization	9056 mod./353.2	4 oz	Soil Jar	1	48 hrs (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest.,Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	28 days (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	9056 mod./365.2	4 oz	Soil Jar	1	48 hrs (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 G	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	SM2540 G, current CLP SOW	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 G	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9038	4 oz	Soil Jar	1	28 days (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Carbon	9060	4 oz	Soil Jar	1	28 days
Total Organic Carbon	Llyod Kahn	4 oz	Soil Jar	1	14 days
Total Organic Carbon	Walkley Black	4 oz	Soil Jar	1	14 days
ELEMENTAL ANALYSES – SOLID					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS ELements	6020	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
GC ORGANIC ANALYSES – SOLID					
Extractable Petroleum Hydrocarbons	MADEP EPH	4 oz	Soil Jar	1	14days/40days

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SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
Fuel Oil	ME HETL 4.1.25 & 8015 mod.	4 oz	Soil Jar	1	14days/40days
Petroleum Range Hydrocarbons	FL-PRO	4 oz	Soil Jar	1	14days/40days
Total Petroleum Hydrocarbons	TX1005	4 oz	Soil Jar	1	14days/14days
Extracted Total Petroleum Hydrocarbons	CT-ETPH	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17 & 8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP VPH	(2)40 mL	VOA vial	1,13	28days
HPLC ANALYSES – SOLID					
HPLC-Explosives	8330B/B Mod.	4 oz or ISM sample	Soil Jar	1	14days/40days
GC/MS ANALYSES – SOLID					
Acid Extractables	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables & (SIM)	8270/8270 SIM	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg) (Please refer to Figure 6-2 for details on collection and preservation)	5035/8260	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2
Volatile Organics – Low Soil (<200 ug/kg) (Please refer to Figure 6-2 for details on collection and preservation)	5035/8260	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2	Please refer to Figure 6-2
Volatile Organics & (limited SIM)	8260/8260 SIM	(2) 40 mL	VOA Vial	1	14 days
Miscellaneous – SOLID					
Grain Size (sieve and hydrometer)	ASTM D422	8 oz	Soil jar or bag	1	none
RCRA - HAZARDOUS WASTE CHARACACTERIZATION					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
TCLP					
TCLP Extraction-Volatile Organics	1311/8260	100 g	Soil Jar	1	14 days/14 days
TCLP Extraction-Semivolatiles	1311/8270	200 g	Soil Jar	1	14 days/7 days/40 days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
TCLP Extraction-Pesticides & Herbicides	1311/8081 & 8151	400 g	Soil Jar	1	14 days/7 days/40 days
TCLP Extraction-Metals	1311/6010/6020	200 g	Soil Jar	1	28 days/180 days
TCLP Extraction-Mercury	1311/7470	200 g	Soil Jar	1	28 days/28 days
GC/MS ANALYSES - AIR					
Volatile Organics	TO-15	(1) 1.4 or 6 L	Canister	16	30 days
Volatile Organics	MA-DEP APH	(1) 1.4 or 6 L	Canister	16	30 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H2SO4 to pH<2
4 = HNO3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M Na2S2O3 or 1 10 mg pellet
7 = 1 m/L 2NZnAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = Na2S2O3, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate
16 = None
17 = benzalkonium chloride

~ Hold time for unpreserved samples is 7 days.

^ Because there are no published holding times for Wet Chemistry soil methods, these are only recommended holding times. They are not regulatory.

Project-specific (i.e. CLP, NYSDEC) hold times take precedence over these hold times as appropriate.

For solid samples, please place parameters of the same analytical group (ie. wet chemistry) in the same container whenever possible. Also, organic and inorganic parameters should be placed in separate containers. Volatile organics should always be placed in organic-free jars. Several 4 oz. soil jars may be needed when numerous parameters are required.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 1

EXAMPLE OF STANDARD KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin ANALYTICAL SERVICES
600 Technology Way
Scarborough, ME 04074
Tel: (207) 874-2400
Fax: (207) 775-4029

CHAIN of CUSTODY

PLEASE BEAR DOWN AND PRINT LEGIBLY IN PEN

Page ____ of ____

Client	Contact	Phone # ( )	Fax # ( )
Address	City	State	Zip Code
Purchase Order #	Proj. Name / No.		Katahdin Quote #
Bill (if different than above)	Address		
Sampler (Print / Sign)			Copies To:

LAB USE ONLY | WORK ORDER #:
KATAHDIN PROJECT NUMBER _____
REMARKS: _____
SHIPPING INFO: ❒ FED EX ❒ UPS ❒ CLIENT
AIRBILL NO: _____
TEMP°C _____ ❒ TEMP BLANK ❒ INTACT ❒ NOT INTACT

ANALYSIS AND CONTAINER TYPE / PRESERVATIVES

*	Sample Description	Date / Time coll'd	Matrix	No. of Cntrs.	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N	Filt. ❒Y ❒N
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COMMENTS

Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)
Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)

THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES, EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF KATAHDIN HOMEOWNER CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Drinking Water Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

***Test results are for compliance and will be reported to the state (see statement below) yes no — Compliance samples may need to be received on ice.

Lab Use Only | Work Order #: | KAS Project Manager:

Shipping: UPS Fed-Ex Mail Drop-Off

Sample(s) Received on Ice? Yes No Temperature if Iced:

Requested Services

Sample Description (Sample Identification and/or Lot #)	Date Collected	Time Collected	No. of Cntrs.	Standard Homeowner	Arsenic	Total Coliforms, e-coli	Lead (1st draw)	Safety Test – coliform,N+N	FHA/MSH	Fluoride	Uranium		What's Included in the Standard Test and the FHA/MSH Test.
													Standard Homeowner: Total Coliform/e-coli, Nitrate, Nitrite, Chloride, pH, Hardness, Uranium, Copper, Iron, Lead, Manganese, Sodium, Arsenic
													FHA/MSH: Standard plus Lead(1st draw), Turbidity, Color, Odor

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

Per the National Environmental Laboratory Accreditation Program (NELAP) Standards, Katahdin is required to accept samples that have been properly preserved. All sample containers provided to you have been properly preserved, but the proper preservation also requires samples to be received at temperatures specified in the regulations. The Safe Drinking Water Act regulations only require this for compliance samples (i.e., results that are submitted to the state). By circling no for compliance (above), you acknowledge that the samples described above are not for compliance purposes, and thus may not meet the temperature receipt requirements. All services shall be governed by Katahdin's standard terms and conditions.

QA-059 – Revision 2 – 03/31/2016

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 3

EXAMPLE OF KATAHDIN FOOD/MICROBIOLOGY CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

Lab Use Only	Work Order #:	KAS Project Manager:	Food & Microbiological Services
Shipping: UPS Fed-Ex	Airbill No.:		
Temperature:			

Sample Description (Sample Identification and/or Lot #)	Date/Time Collected	Matrix	No. of Cntrs.	Plate Count (AHS)	Listeria	Yeast and Mold	Salmonella	E-Coli	E-Coli 0157:H7	Staph	Vibrio	Total Coliforms	Campylobacter	Shelf Life	Challenge Study		

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

The terms and conditions on the following page hereof shall govern services, except when a signed contractual agreement exists.

QA-058 – Revision 1 – 09/22/2010

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN AIR CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Air Analysis Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

Lab Use Only	Work Order #:	KAS Project Manager:	Requested Services
Shipping:	UPS	Fed-Ex	Mail	Drop-Off	

Sample Description (Sample Identification and/or Lot #)	Collection					Matrix	Sampler	Can Size	Can ID	Flow Controller ID				Comments
	Date	Start Time	End Time	Initial Vac	Final Vac									

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

Katahdin inspects and verifies all equipment including, but not limited to, canisters and flow controllers before being sent to the client. As the client you have agreed to pay a rental fee for use of this equipment, which is the sole property of Katahdin. All equipment will be inspected for damage and completeness upon return to Katahdin. In the event that rental equipment is missing and/or damaged, by signing this COC, you (the client) agrees to pay Katahdin for replacement of any unuseuable, damaged or missing equipment.

QA-132 – Revision 1 – 02/06/2014

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBOOK

KATAHDIN ANALYTICAL SERVICES, LLC.
SAMPLE LOG IN

pH Paper Lot #: 7K 564756

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
5/13/16	1345	5-13-16	1400	SJ 3311	Harmon's	
5/13/16	1350	5-13-16	15:00	SJ 3312	Bristol Seafood	
↓	↓			SJ 3313	Camp Sunshine	↓
5-13-16	14:00			SJ 3314	DEP-B	
				SJ 3315	↓	
				SJ 3316	DEP-A	
				SJ 3317	↓	
			15:30	SJ 3318	FGS	
				SJ 3319	CES	
				SJ 3320	↓	
				SJ 3321		
			↓	SJ 3322	SWCole	
		5-13-16	16:00	SJ 3323	Clearwater	
				SJ 3324		
				SJ 3325		
				SJ 3326		
				SJ 3327		
				SJ 3328		
				SJ 3329		
				SJ 3330		
				SJ 3331		
				SJ 3332		
				SJ 3333		
		↓	↓	SJ 3334	PWD	↓ open
↓	↓	5-13-16	16:40	SJ 3335	Cape Elizabeth Tax	
5-13-16	15:00			SJ 3336	MEL	
↓	↓			SJ 3337	↓	↓
5-13-16	15:35			SJ 3338	PWD	
5/13/16	1540			SJ 3339	Maine Medical	
				SJ 3340	COLM	
↓	↓	↓	↓	SJ 3341	↓	↓

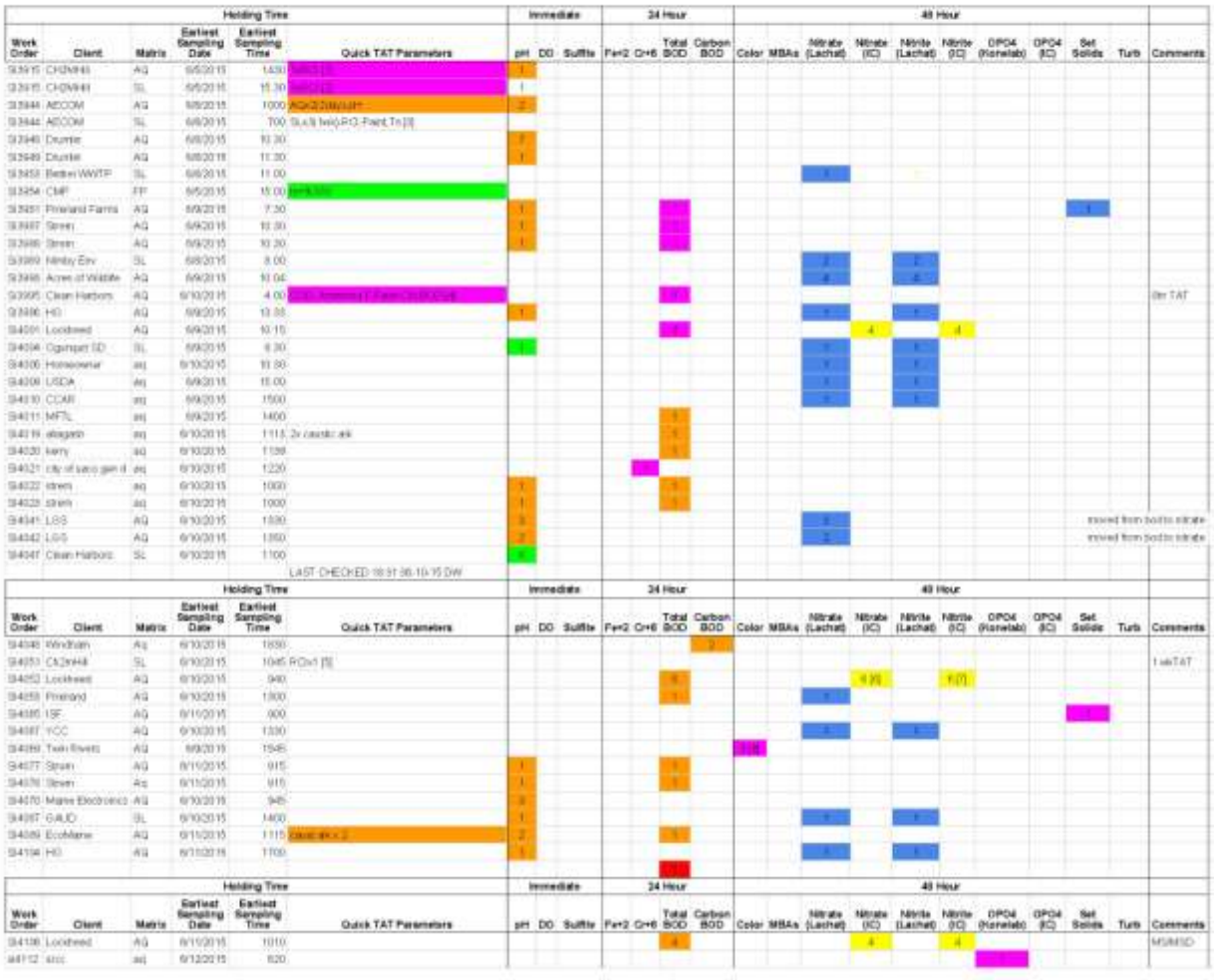
QA-032 - Revision 1 - 12/30/2009 Updated: 04/26/2016

QAQC793 0000011

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES SCREEN SHOT



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 7

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

Katahdin Analytical Services, LLC. **Sample Receipt Condition Report**

Client:	KAS PM:	Sampled By:
Project:	KIMS Entry By:	Delivered By:
KAS Work Order#:	KIMS Review By:	Received By:
SDG #:	Cooler: _____ of _____	Date/Time Rec:

Receipt Criteria	Y	N	EX*	NA	Comments and/or Resolution
1. Custody seals present / intact?					
2. Chain of Custody present in cooler?					
3. Chain of Custody signed by client?					
4. Chain of Custody matches samples?					
5. Temperature Blanks present? If not, take temperature of any sample w/ IR gun.					Temp (°C):
Samples received at <6 °C w/o freezing?					Note: Not required for metals (except Hg soil) analysis
Ice packs or ice present?					The lack of ice or ice packs (i.e. no attempt to begin cooling process) or insufficient ice may not meet certain regulatory requirements and may invalidate certain data.
If yes, was there sufficient ice to meet temperature requirements?					
If temp. out, has the cooling process begun (i.e. ice or packs present) and sample collection times <6hrs., but samples are not yet cool?					Note: No cooling process required for metals (except Hg soil) analysis.
6. Volatiles: **Aqueous:** No bubble larger than a pea? **Soil/Sediment:** Received in airtight container? Received in methanol? Methanol covering soil? D.I. Water - Received within 48 hour HT?					
Air: Refer to KAS COC for canister/flow controller requirements.	√ if air included				
7. Trip Blank present in cooler?					
8. Proper sample containers and volume?					
9. Samples within hold time upon receipt?					
10. Aqueous samples properly preserved? Metals, COD, NH3, TKN, O/G, phenol, TPO4, N+N, TOC, DRO, TPH – pH <2 Sulfide - >9 Cyanide – pH >12					

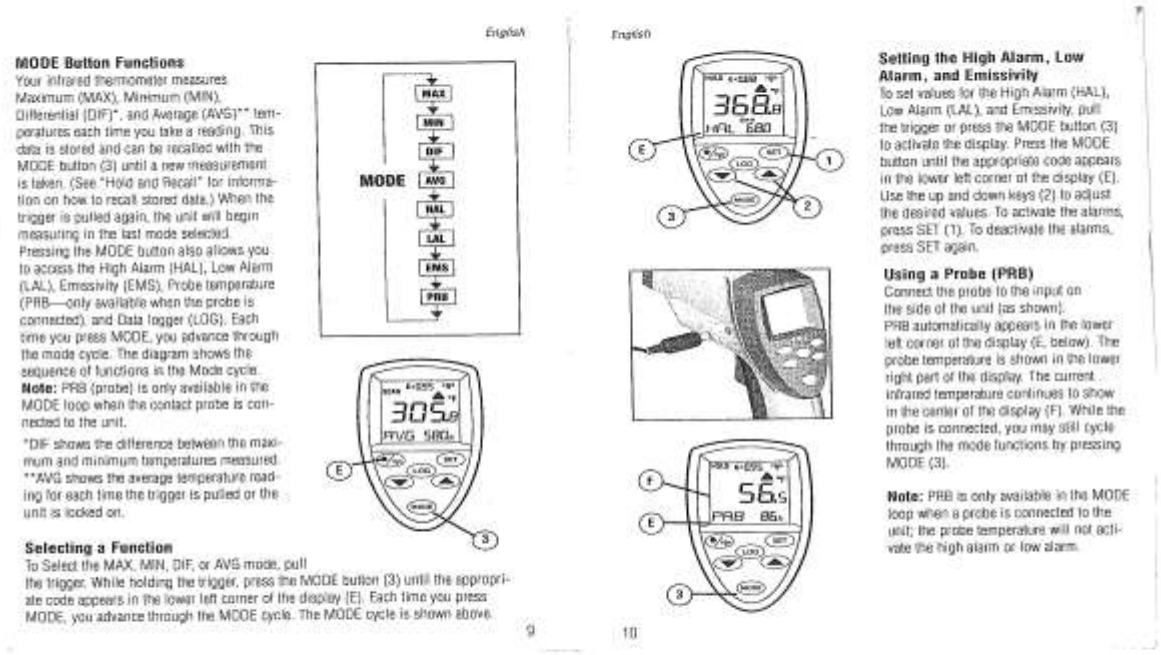
* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments.

QA-048 – Revision 6 – 07/20/2015

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8

IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY



English

MODE Button Functions

Your infrared thermometer measures Maximum (MAX), Minimum (MIN), Differential (DIF)*, and Average (AVG)** temperatures each time you take a reading. This data is stored and can be recalled with the MODE button (3) until a new measurement is taken. (See "Hold and Recall" for information on how to recall stored data.) When the trigger is pulled again, the unit will begin measuring in the last mode selected. Pressing the MODE button also allows you to access the High Alarm (HAL), Low Alarm (LAL), Emissivity (EMS), Probe temperature (PRB—only available when the probe is connected), and Data logger (LOG). Each time you press MODE, you advance through the mode cycle. The diagram shows the sequence of functions in the Mode cycle.

Note: PRB (probe) is only available in the MODE loop when the contact probe is connected to the unit.

*DIF shows the difference between the maximum and minimum temperatures measured.
**AVG shows the average temperature reading for each time the trigger is pulled or the unit is locked on.

Selecting a Function

To Select the MAX, MIN, DIF, or AVG mode, pull the trigger. While holding the trigger, press the MODE button (3) until the appropriate code appears in the lower left corner of the display (E). Each time you press MODE, you advance through the MODE cycle. The MODE cycle is shown above.

9

English

Setting the High Alarm, Low Alarm, and Emissivity

To set values for the High Alarm (HAL), Low Alarm (LAL), and Emissivity, pull the trigger or press the MODE button (3) to activate the display. Press the MODE button until the appropriate code appears in the lower left corner of the display (E). Use the up and down keys (2) to adjust the desired values. To activate the alarms, press SET (1). To deactivate the alarms, press SET again.

Using a Probe (PRB)

Connect the probe to the input on the side of the unit (as shown). PRB automatically appears in the lower left corner of the display (E, below). The probe temperature is shown in the lower right part of the display. The current infrared temperature continues to show in the center of the display (F). While the probe is connected, you may still cycle through the mode functions by pressing MODE (3).

Note: PRB is only available in the MODE loop when a probe is connected to the unit; the probe temperature will not activate the high alarm or low alarm.

10

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Sample Filtration Logbook

Katahdin Sample No. List Individually	Site ID (Optional)	Filtration Requested By:		Filtered and Preserved By:		
		Initials	Date	Initials	Date	Time

Reviewed and Approved by: _____ **Date:** _____

ME-005 - Revision 1 - 09/23/2010

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 10

MEASURED TURBIDITY AND PRESERVATION OF INCOMING SAMPLES LOGBOOK

KATAHDIN ANALYTICAL SERVICES

Measured Turbidity and Preservation of Incoming Samples

KAS Lab Sample ID	Measured Turbidity (NTU)	Turbidity Date	Turbidity Analyst	Preservation Date	Preservation Time	Preservation Analyst
REVIEWED BY:			DATE:			

QA-068 – Revision 1 – 09/23/2010

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF KIMS LABORATORY INCOMING SAMPLE REPORT

Katahdin ANALYTICAL SERVICES

INCOMING SAMPLE REPORT

Quote: ELM001 Account: ELM001 Project:
Company: [redacted] Quote Date: 29-MAR-10 Expires:
Name: [redacted] Date Expected: 28-SEP-40
Address: [redacted] Email: [redacted]

Notes: 8-aqueous VOA, 4-MEE,22-TOC, 44-metals (Total & Dissolved), 22-alkalinity, 12-Chloride,Nitrate, Nitrite, Sulfate

Analysis Notes: Merge Results for EDD

Report Notes: Email pdf and EDD to maltmayer@elmllc.com and tsnarr@elmllc.com, Mail rpt and EDD on CD, no HC, metals need to be rpt mg/l. Down load rpt to FTP site for ELM. Hold Rpt Till Payment See Daphne

Description:
Project Name: Pilot Test [redacted] Client PO:
QCLevel: II Tat: 13 Terms: Reg List: Edd: KAS064-XLS

Product	Matrix	Quant	STD or Special Lists	Short	Unit Price	Total Price
E325.2-CHLORIDE	AQ	1	STD		40	40
E353.2-NITRATE	AQ	1	STD	SHORT	0	0
E353.2-NITRITE	AQ	1	STD	SHORT	0	0
E375.4-SULFATE	AQ	1	STD		0	0
RSKSOP175-MEE	AQ	1	STD		85	85
SM5310B-TOC	AQ	1	STD		25	25
SW3010-PREP	AQ	1	STD		0	0
SW6010-ARSENIC	AQ	1	STD		60	60
SW6010-ARSENIC-DIS	AQ	1	STD		60	60
SW6010-CALCIUM	AQ	1	STD		0	0
SW6010-CALCIUM-DIS	AQ	1	STD		0	0
SW6010-IRON	AQ	1	STD		0	0
SW6010-IRON-DIS	AQ	1	STD		0	0
SW6010-MAGNESIUM	AQ	1	STD		0	0
SW6010-MAGNESIUM-DIS	AQ	1	STD		0	0
SW6010-MANGANESE	AQ	1	STD		0	0
SW6010-MANGANESE-DIS	AQ	1	STD		0	0
SW6010-POTASSIUM	AQ	1	STD		0	0
SW6010-POTASSIUM-DIS	AQ	1	STD		0	0
SW6010-SODIUM	AQ	1	STD		0	0
SW6010-SODIUM-DIS	AQ	1	STD		0	0
SW8260FULL_LD	AQ	1	STD		115	115
						$ 385.00

History: _____
Other: _____
_____

Printed: 21-SEP-10 1 of 1

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services

Login Chain of Custody Report (lno1)

Jan. 26, 2007
03:51 PM

Page: 1 of 1

Login Number: SA0395
Account:KATAHD001
Katahdin Analytical Services
Project:

Web

Login Information

ANALYSIS INSTRUCTIONS	:
CHECK NO.	:
CLIENT PO#	:
COOLER TEMPERATURE	: n/a
DELIVERY SERVICES	: In House
EDD FORMAT	:
MAIL DATE	:
PM	: LAD
PROJECT NAME	: QC Holding Blanks
QC LEVEL	: I
REGULATORY LIST	:
REPORT INSTRUCTIONS	:
SDG ID	:
SDG STATUS	:

Primary Report Address:
Leslie Dimond
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Primary Invoice Address:
Accounts Payable
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Report CC Addresses:
Invoice CC Addresses:

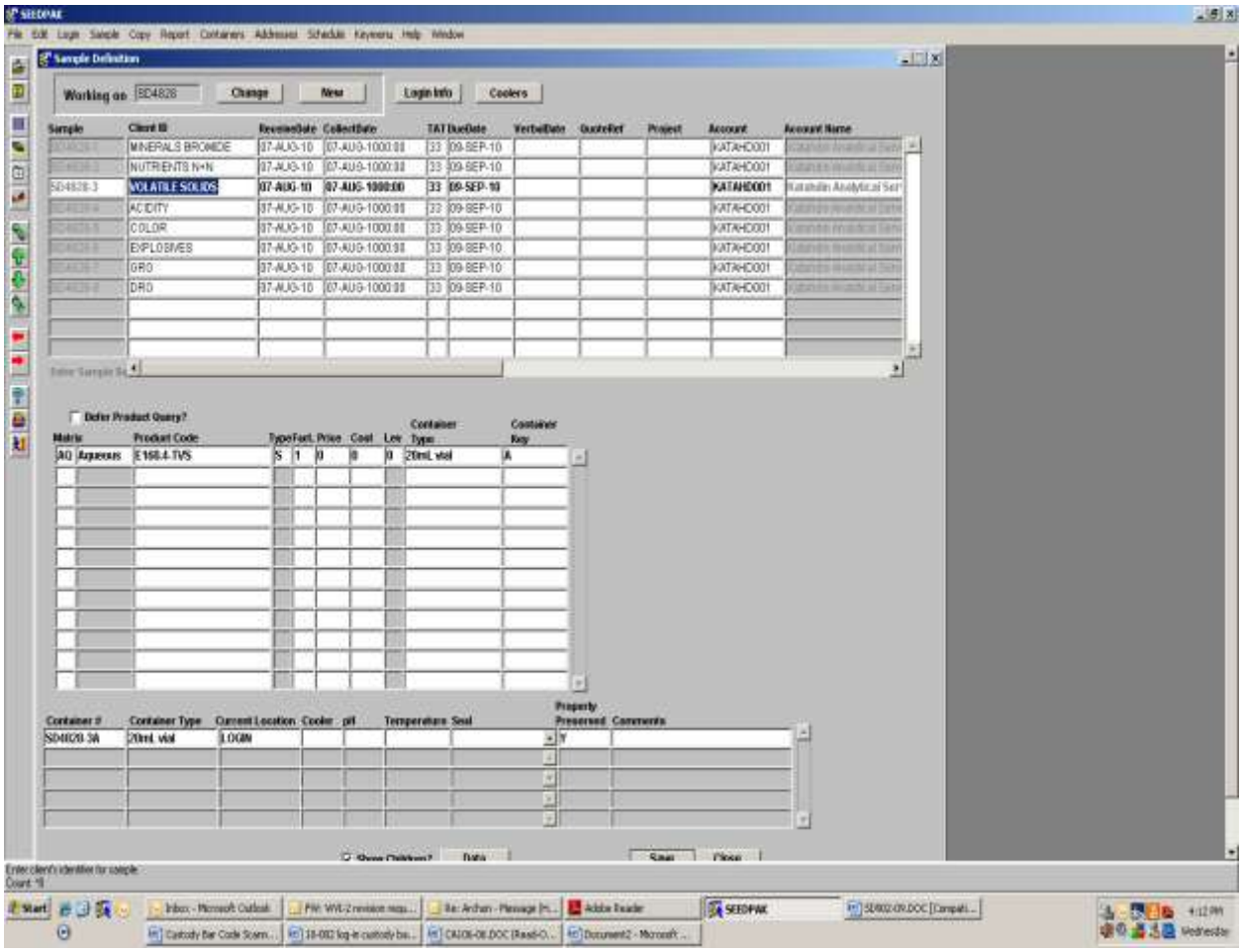
Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULLMS	09-FEB-07			3		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULLMS	09-FEB-07			3		

Total Samples: 2 ***Total Analyses:*** 2

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF LOGIN SCREEN IN KIMS



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 14

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

Sample Receipt Refrigerator and Freezer Temperature Logbook

Corrective Action: If acceptance criteria are not met, notify the QAO or your supervisor immediately to determine corrective action to be taken. Document the corrective action in the Comments section.

Thermometer Location		Sample Receipt Refrigerator 1	Sample Receipt Freezer 1	Comments
Acceptance Criteria		Above 0 to 6 °C	< -10 °C	
Date	Initials	Temp (°C)	Temp (°C)	

QA-069 – Revision 1 – 06/12/2013

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 15

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

QA-046 – Revision 1 – 04/15/2010

KATAHDIN ANALYTICAL SERVICES, INC.
INTERNAL CUSTODY RECORD FOR IMMEDIATES

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no
							yes no

0000001

FIGURE 16

SAMPLE ACCEPTANCE POLICY

Katahdin Analytical Services Sample Acceptance Policy

Katahdin Analytical Services reserves the right to refuse any samples due to any anomalies, discrepancies or non-compliances concerning the receipt and/or analysis of samples. These may include but are not limited to:

- Insufficient sample volume
- Insufficient remaining holding time
- Health or safety risks the samples may pose, including radioactivity
- Insufficient experience to handle sample or analysis
- Improper or illegible labeling of samples
- Improper sample containers
- Insufficient documentation including sample identification, location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample
- Damaged, contaminated or inadequately preserved samples

Any decisions to reject samples are made with the client's input.

TITLE: **SAMPLE DISPOSAL**

Prepared By: _____ Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: _____ Date: 2/01

QA Officer: _____ Date: 2.01

General Manager: _____ Date: 2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, ~~added pollution prevention~~, added updated log book and greater detail on disposal.	DH	2.01	2/01
02	Major rewrite to include more detail on hazardous waste regulations & to reflect current practices.	DH	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	DH	02.08	02.08
04	Added elementary neutralization to section 7.0. Other minor edits.	DH	05.09	0509
05	Sect. 7 - Added non-hazardous samples are recycled, added PCB information, changed elementary neutralization target pH to 5-~~8~~9. Added wording for clarification. Updated Figures 1, 3 and 5.	LAM	06/13	06/13

TITLE: SAMPLE DISPOSAL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-903-05**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-903-05**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

TITLE: SAMPLE DISPOSAL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires strict adherence to specific procedures for the disposal of samples. The procedures are designed to categorize waste materials, provide for their safe and timely disposal and to ensure compliance with local and federal regulations pertaining to disposal of chemicals and environmental samples. Any other means of disposal not described in this SOP is prohibited without consent from the Katahdin Environmental Health & Safety Officer and/or the Katahdin Environmental Compliance Officer.

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical personnel for the disposal of samples. These procedures apply to the disposal of all samples received or processed by Katahdin. Refer to the current revision of Katahdin SOP CA-107 regarding the disposal of spent preparation and analysis reagents, standards, sample extracts, distillates, or digestates.

1.1 Definitions

Hazardous Waste – A “Solid Waste” which displays a hazardous characteristic or is specifically listed as hazardous waste.

Solid Waste – Any discarded material that is not excluded from the definition of hazardous waste.

Discarded Material – Material that is abandoned, recycled or inherently waste-like.

Waste (State of Maine) –

- Any useless, unwanted, or discarded substance or material, whether or not such substance or material has any other future use.
- Any substance or material that is spilled, leaked, pumped, poured, emptied or dumped onto the land or into the water or ambient air.
- Materials which are used in a matter constituting disposal, burned for energy recovery, reclaimed, or accumulated speculatively.

Ignitable Hazardous Waste – EPA Waste Code D001

- Liquids with a flash point less than 140°F or 60°C.
- Solids capable of spontaneous combustion under normal temperature and pressure.
- Ignitable compressed gas.
- Oxidizers.

Corrosive Hazardous Waste - Liquids with a pH less than or equal to 2.0 or greater than or equal to 12.5. EPA waste code D002.

Reactive Hazardous Waste – EPA waste code D003.

- A material that reacts violently with water.
- A material that generates toxic gases or fumes.
- Explosives.

Toxic Hazardous Waste – A material that exceeds certain concentration levels based on the toxicity characteristic leaching procedure (TCLP). See Figure 3 for the chemicals and concentration levels covered under this definition.

Listed Wastes – Lists of chemicals that are considered hazardous based on the following criteria

- Virgin chemical or unused product.
- Sole active ingredient.
- Single substance spill debris.

Listed wastes are divided into 5 subcategories

- F-wastes – Describe hazardous waste from non-specific sources usually containing halogenated and non-halogenated solvents.
- K-wastes – Describe hazardous wastes created by specific processes.
- U-wastes – Describe toxic or non-acute hazardous wastes.
- P-wastes – Describe acute hazardous wastes. (Note: Maine considers a material to be a P-listed waste if it contains 10% or more of any P-listed chemical.
- State listed wastes – Maine lists any material with a concentration of greater than 50 ppm Polychlorinated Biphenyls (PCB) as a hazardous waste.

Organics hit – A liquid sample containing greater than 1 mg/L of organic contaminants or a soil sample containing greater than 20 mg/kg of organic contaminants.

1.2 Responsibilities

Only designated analysts/technicians trained in these procedures may dispose of samples or analytical by-products. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Gloves, safety glasses, lab coats and/or other protective clothing must be worn at all times.

It is the responsibility of the designated Katahdin personnel involved in the disposal of samples to read and understand this SOP, to adhere to the procedures outlined, to properly document their activities in the appropriate lab notebook and file the necessary manifests and reports to outside agencies in the required manner. Refer to

Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of the Department Managers to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

It is the responsibility of the Katahdin Environmental Health & Safety Officer (EHSO) to manage the proper classification and disposal of samples. Katahdin is responsible for regulatory compliance of Katahdin's waste storage areas (less than 90 day storage). The EHSO ensures compliance of the waste storage areas with applicable state and federal regulations. The EHSO is responsible for providing the appropriate training to all individuals involved in the proper classification and/or disposal of samples. The EHSO is responsible for working with the Laboratory Operations Manager/Environmental Compliance Officer to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate unresolved problems and concerns to the Laboratory Vice President.

It is the responsibility of the Operations Manager/Environmental Compliance Officer to oversee adherence to Katahdin sample disposal and hazardous waste practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the EHSO and/or the Laboratory Vice President.

It is the responsibility of the Laboratory Vice President to provide the necessary resources to meet the regulatory requirements of proper classification and disposal of samples.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

Not applicable.

TITLE: SAMPLE DISPOSAL

5.0 REAGENTS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

7.1 Sample purging is the removal of samples from laboratory refrigerated storage. Sample storage areas where samples are removed (purged) from include wet chemistry, organic extractables, metals, volatiles, total organic carbon and soils. Wet chemistry, aqueous metals, organic extractables, total organic carbon, and soils can all be found in the walk-in refrigerator. Aqueous and soil volatiles can be found in the volatiles laboratory refrigerators/freezer.

7.2 Samples are purged from storage, after analysis and reporting, on a routine basis to make room for incoming samples. Samples are to be kept in storage for a duration of 30 days past the report mailed date. Some samples must be kept for 60 or 90 days beyond the report mailed date, depending on specific client requests and contracts.

7.3 The first step in disposing of samples is to generate a disposal list. The disposal list contains sample analysis information stored in the Katahdin Information Management System (KIMS). The analytical data for the samples is compared to the hazardous waste criteria specified in 40CFR Part 261 and to local wastewater discharge criteria. Refer to Figure 4 for 40 CFR Part 261 Characteristic Hazardous Waste Criteria. Based on this comparison, the report displays information on the classification/category for disposal of each sample. The disposal report should be reviewed against the data reports for accuracy. Refer to Figure 2 for an example of a KIMS generated disposal list. The primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide. Katahdin has established 14 waste stream profiles with a 3rd party waste transporter/waste disposal firm for sample disposal based on these categories. As required, new or special temporary waste profiles are established based on the characteristics of samples.

7.4 Sorting through samples and preparing them for disposal is a crucial quality checkpoint. Samples put into the incorrect waste stream could not only produce adverse environmental effects, but, could also interrupt the 3rd party's waste treatment efficiency, or endanger an individual handling the waste stream. Therefore, when sorting through samples pay close attention to which waste stream each sample falls into.

7.5 Once you are ready to dispose of the samples of interest (the oldest samples that have been purged), these samples must be sorted, logged, and the classification/category (sample knowledge) information recorded.

Sample storage times (as listed in section 7.2) and space should be taken into consideration when purging samples. It is important to make room for future samples, but to make sure that samples are not purged too early. Samples should be pulled from the walk-in or the volatiles refrigerators to make room for new samples. When purging, chose a section that needs extra space the most and remove the oldest samples.

Safety glasses, nitrile gloves, lab coat, and a splash apron must be worn when handing samples during disposal

7.6 Remove the designated purge samples from the shelf one by one and line them up on the countertop in the log-in area. Generally, removing two cartloads at a time is a good amount to purge at one time. For volatile samples in 40mL vials, 5 or 6 vial trays should be purged at a time. Samples should be lined up across the counter with the earliest sample to the left and building up to the right, organizing the samples according to work order and sample number. After the samples are lined up, they should be recorded in the Sample Disposal Logbook (SDL). Refer to Figure 1 for an example SDL page. The location the samples were removed from should also be recorded. Sample storage areas are recorded with the following designations:

VOA (Aq)	Aqueous Volatiles (VOA)
VOA (SL)	Solid Volatiles (VOA)
M	Metals
EXT	Extractables (Organic)
TOC	Total Organic Carbon
WC	Wet Chemistry
S	Soils

7.7 The next step is to use the sample disposal list to determine the earliest release date of the reports and to determine each samples appropriate waste classification/characterization. As stated in section 7.3, the primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide.

Using the information from the KIMS disposal list, record the appropriate classification for each sample in the SDL. If multiple categories are identified as being present then a single category is selected as controlling. The order of precedence is PCB's, metals and then organics. If another scenario is found, the individual should bring it to the EHSO for a determination of the acceptable waste stream designation or a determination that it should be lab packed separately.

If samples have been sorted that have not been in storage for the 30 days beyond the release date (60 or 90 for certain clients), then these samples need to be placed back in storage and it should be noted in the SDL.

7.8 As stated above, a sample may be categorized into a waste stream based upon the analytes it contains as determined by laboratory testing. In addition, many samples are also categorized as hazardous waste based upon the preservative that they contain. Since many samples contain preservatives, caution must be used when dumping samples. It is also important to ensure that the sample container is empty. This can be accomplished by holding the container upside down and shaking gently until liquid is no longer observed coming out of the container.

7.9 Once waste categories have been determined and entered into the SDL, The following waste categories are disposed of as follows:

7.9.1 Dumping non-hazardous samples (as determined by laboratory testing)

Non-hazardous liquid samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples and their containers are disposed of with the recycling trash, which is picked up by commercial trash collectors and ultimately turned into construction material.

7.9.2 Dumping Samples with high Organics (as determined by laboratory testing)

Aqueous samples get dumped into waste stream “K”. Containers are disposed of with general trash. Solid samples are placed into waste stream “I” with their containers. The disposal date is recorded in the SDL.

7.9.3 Dumping samples high in metals, including mercury (as determined by the by laboratory testing)

Aqueous samples get disposed of in waste stream “A”. Containers are disposed of with general trash. Solid samples are placed in waste stream “L” with their containers. The disposal date is recorded in the SDL.

7.9.4 Dumping Acidic Samples that do not contain any other hazardous waste constituents (as determined by the acidic preservative or by laboratory testing)
Refer to section 7.10 below.

7.9.5 Dumping samples with high PCBs (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “Q”. Containers are disposed of with general trash. Solid samples get disposed of in waste stream “F” with their containers. The disposal date is recorded in the SDL. Any PCB samples with PCB content 50 ppm or greater, solid or aqueous, are set aside for TCSA regulated disposal.

7.9.6 Dumping samples with low flashpoints (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “O”. Containers are disposed of with general trash. Solid samples get disposed of in waste stream “I” with their containers. The disposal date is recorded in the SDL.

7.9.7 Dumping samples with high cyanide (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream “NHi”. Containers are disposed of with general trash. Solid samples should be set aside for labpack. The disposal date is recorded in the SDL.

7.9.8 Miscellaneous Disposal (as determined by the preservative)

7.9.8.1 Sodium Bisulfate: Sodium Bisulfate often comes in vials, but may also come in the 2-4oz glass jars. Dump the Sodium Bisulfate out of the container into waste stream “A”. There may be remaining soil left in the sample container. The soil’s waste stream and dump date will be dictated by the SDL. The disposal date is recorded in the SDL.

7.9.8.2 Methanol / Free Products: This often comes in vials, but may also come in the 2-4oz glass jars. Dump the methanol out of the container into the mix-flammables accumulation. When this satellite accumulation container gets full it can be dumped into the “O” waste stream. There may be remaining soil left in the sample container. The soil’s waste stream and dump date will be dictated by the SDL. Lastly, samples marked “free product” on the Katahdin sample ID label can be dumped into the mixed flammables stream. The disposal date is recorded in the SDL.

7.10 Pursuant to Maine DEP regulations, Katahdin has the necessary agreements, processes and documentation in place to neutralize samples without a license. Refer to the current revision of the Katahdin Environmental Health & Safety Manual for additional information. Generally, the following procedures are followed.

7.10.1 Samples that have been determined to be hazardous due **solely** to the corrosivity characteristic are neutralized using sodium hydroxide pellets. In the warehouse, samples are emptied into a five gallon heavy duty carboy to about 60% capacity. The carboy is kept in a secondary container. Sodium

hydroxide pellets are added slowly to the carboy (about 5 grams at a time) and stirred with a long glass stirring rod. The pH is checked with pH paper.

7.10.2 This process is continued until the pH is between 5 and 9. This normally takes about 30-40 grams of sodium hydroxide pellets, but may vary depending on the buffering capacity of the individual samples.

7.10.3 The carboy is emptied into the sink in the warehouse. The tap water is run at the same time as the neutralized material is disposed of. An eyewash station and spill material is located at this sink.

7.10.4 All neutralization activities are documented, including the date and time of neutralization, the name of the person doing the neutralizing, the amount of neutralized liquid discharged, details on the inspection of the drain area and the date and nature of any significant repairs or corrective actions. This documentation is maintained by the EHSO. Refer to Figure 5 for an example logbook page of neutralization documentation.

7.11 Dumping Basic samples (as determined by the basic preservative or by laboratory testing). If the samples have been to be hazardous due solely to the corrosivity characteristic, they are included in the neutralization process above.

7.12 Every 3 to 5 weeks a pickup of hazardous waste is scheduled with the 3rd party waste transporter/waste disposal firm. An inventory is faxed to the transporter summarizing the number of drums and waste streams/profiles. As required, a “lab pack” of expired chemicals or orphan samples is organized as necessary. A designated individual, with applicable Hazardous Waste (RCRA) and Department of Transportation (DOT) training, oversees the waste pickup and signs the hazardous manifests and land ban documentation. Within 7 days a copy is forwarded to the Maine Department of Environmental Protection (MEDEP) and the environmental agency in the designation state (if required by that state). Once the report is received at the disposal facility a copy is returned to KATAHDIN and the MEDEP.

7.13 Prior to March 31 of each year, the laboratory prepares the Annual Hazardous Waste Report (i.e., MEDEP modified EPA Form 8700-13A) as required by MEDEP Hazardous Waste Management Rules. The complete report is reviewed by the Katahdin Environmental Compliance Officer and then forwarded to the following address:

Maine Department of Environmental Protection
Bureau of Remediation & Waste Management
State House Station #17
Augusta, ME. 04333
Attn: Annual Hazardous Waste Report

TITLE: SAMPLE DISPOSAL

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

On a daily basis, a designated individual performs quality checks in all hazardous waste storage areas. The daily check documentation is located in login. Any discrepancy is copied to the Operations Manager and the Katahdin Vice President for corrective action. Refer to the current revision of Katahdin SOP CA-107, *The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents & Standards*, for more information. Refer to Figure 3 for a copy of the daily check documentation.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Code of Federal Regulations, 40 CFR Part 261.

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

LIST OF TABLES AND FIGURES

TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

QA-022 - Revision 1 - 11/09/2009

QAQC645

0000012

KATAHDIN ANALYTICAL SERVICES, INC. –SAMPLE STORAGE/DISPOSAL LOGBOOK

WORK ORDER/ SAMPLE NUMBERS	DEPARTMENT	EARLIEST RELEASE DATE	< CRITERIA	SAMPLE KNOWLEDGE									DATE DISPOSED	INITIALS
			CLEAN	WL	ORG	METS	CN	FP	HG	PCBS				
SG-1969-1	M	4-10-13		✓					✓				5-20-13	5-20-13
SG-1970-1		4-9-13		✓										
SG-1977-1		4-4-13	✓											
SG-1978-1-22		4-18-13	1-10, 12-H 16-22		11, 15									
SG-1979-2-10		4-10-13	✓											
SG-1990-1		4-4-13				✓								
SG-1991-1	↓	4-4-13				✓							↓	↓
SG-1002-1	WC	2-27-13	✓										5-21-13	5-21-13
SG-1010-1		2-26-13	✓											
SG-1017-1		2-21-13	✓											
SG-1021-1		2-26-13	✓											
SG-1022-1,2	↓	2-28-13	2	1									↓	↓

TITLE: SAMPLE DISPOSAL

FIGURE 2

EXAMPLE OF KIMS GENERATED WASTE DISPOSAL REPORT

SAMPLE DISPOSAL REPORT

Query by: Login SA6501 to SA7000
Date : 15-JAN-08

Sample	SDG	Status	Mail Date	Parameter	Value		
SA6605-1		NEED	12/02/07				
SA6606-1		NEED	12/02/07				
SA6607-1		NEED	11/15/07				
SA6608-1		NEED	12/06/07				
				ORG	1.17	MG/L	(HIGH)
SA6608-1		NEED	12/06/07				
SA6608-2		NEED	12/06/07				
				AA	13	MG/KG	(HIGH)
SA6609-1		NEED	11/26/07				
SA6609-1		NEED	11/26/07				
SA6610-1		NEED	11/30/07				
SA6611-1	FCS-020	NEED	12/07/07				
SA6611-2	FCS-020	NEED	12/07/07				
SA6611-3	FCS-020	NEED	12/07/07				
SA6611-4	FCS-020	NEED	12/07/07				
SA6611-5	FCS-020	NEED	12/07/07				
SA6611-6	FCS-020	NEED	12/07/07				
SA6611-7	FCS-020	NEED	12/07/07				
SA6611-8	FCS-020	NEED	12/07/07				
SA6612-1	NSA-030	NEED	12/07/07				
SA6612-2	NSA-030	NEED	12/07/07				
SA6612-3	NSA-030	NEED	12/07/07				
SA6612-4	NSA-030	NEED	12/07/07				
				ORG	1.70735	MG/L	(HIGH)
SA6612-5	NSA-030	NEED	12/07/07				
				ORG	1.0481	MG/L	(HIGH)

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TITLE: SAMPLE DISPOSAL

FIGURE 3

EXAMPLE OF HAZARDOUS WASTE STORAGE AREA DAILY CHECK

HZ-007 – Revision 1 – 09/30/2010

Daily Checklist for
HAZARDOUS WASTE STORAGE AREA

Week of: 6-10, 2013

Item/Day:	Monday	Tuesday	Wednesday	Thursday	Friday
1. Are containers closed? (Except when waste is being added)	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
2. Are containers properly labeled with a hazardous waste label?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
3. Do you have access to each container and can you read the label? (36" aisle?)	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
4. Is each container marked with the date storage began?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
5. Are the dates on the containers less than 90 days old?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
6. Is container free of dents, bulges, rust, spills or leaks?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
7. Are all containers on a firm working surface?	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No	(Yes) / No
8. Inspection by: Name (No Initials)					
9. Time of Inspection	14:45	15:10	16:30	12:00	16:00
10. Verification of Inspection (Name/Date)	Dave Medler 6/10/13	Dave Medler 6/11/13	Dave Medler 6/12/13	Dave Medler 6/13/13	Dave Medler 6/14/13

Deficiency noted:					
Corrective action:					
By (Name/Date):					

0000040

TITLE: SAMPLE DISPOSAL

FIGURE 4

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
Arsenic	7440-38-2	D004	5.0 mg/L	100 mg/kg
Barium	7440-39-3	D005	100 mg/L	2000 mg/kg
Cadmium	7440-43-9	D006	1.0 mg/L	20 mg/kg
Chromium	7440-47-3	D007	5.0 mg/L	100 mg/kg
Lead	7439-92-1	D008	5.0 mg/L	100 mg/kg
Mercury	7439-97-6	D009	0.2 mg/L	4 mg/kg
Selenium	7782-49-2	D010	1.0 mg/L	100 mg/kg
Silver	7440-22-4	D011	5.0 mg/L	20 mg/kg
Endrin	72-20-8	D012	0.02 mg/L	0.4 mg/kg
Lindane	58-89-9	D013	0.4 mg/L	8 mg/kg
Methoxychlor	72-43-5	D014	10 mg/L	200 mg/kg
Toxaphene	8001-35-2	D015	0.5 mg/L	10 mg/kg
2,4-D	94-75-7	D016	10 mg/L	200 mg/kg
2,4,5-TP (Silvex)	93-72-1	D017	1.0 mg/L	20 mg/kg
Benzene	71-43-2	D018	0.5 mg/L	10 mg/kg
Carbon Tetrachloride	56-23-5	D019	0.5 mg/L	10 mg/kg
Chlordane	57-74-9	D020	0.03 mg/L	0.6 mg/kg
Chlorobenzene	108-90-7	D021	100 mg/L	2000 mg/kg
Chloroform	67-66-3	D022	6.0 mg/L	120 mg/kg
o-Cresol	95-48-7	D023	200 mg/L	4000 mg/kg
m-Cresol	108-39-4	D024	200 mg/L	4000 mg/kg
p-Cresol	106-44-5	D025	200 mg/L	4000 mg/kg
Cresol	1319-77-3	D026	200 mg/L	4000 mg/kg
1,4-Dichlorobenzene	106-46-7	D027	7.5 mg/L	150 mg/kg
1,2-Dichloroethane	107-06-2	D028	0.5 mg/L	10 mg/kg
1,1-Dichloroethylene	75-35-4	D029	0.7 mg/L	14 mg/kg
2,4-Dinitrotoluene	121-14-2	D030	0.13 mg/L	2.6 mg/kg
Heptachlor	76-44-8	D031	0.008 mg/L	0.16 mg/kg
Hexachlorobenzene	118-74-1	D032	0.13 mg/L	2.6 mg/kg
Hexachlorobutadiene	87-68-3	D033	0.5 mg/L	10 mg/kg
Hexachloroethane	67-72-1	D034	3.0 mg/L	60 mg/kg
Methyl Ethyl Ketone	78-93-3	D035	200 mg/L	4000 mg/kg
Nitrobenzene	98-95-3	D036	2.0 mg/L	40 mg/kg
Pentachlorophenol	87-86-5	D037	100 mg/L	2000 mg/kg
Pyridine	110-86-1	D038	5.0 mg/L	100 mg/kg
Tetrachloroethylene	127-18-4	D039	0.7 mg/L	14 mg/kg
Trichloroethylene	79-01-6	D040	0.5 mg/L	10 mg/kg
2,4,5-Trichlorophenol	95-95-4	D041	400 mg/L	8000 mg/kg
2,4,6-Trichlorophenol	88-06-2	D042	2.0 mg/L	40 mg/kg
Vinyl Chloride	75-01-4	D043	0.2 mg/L	4.0 mg/kg

TITLE: SAMPLE DISPOSAL

FIGURE 5

EXAMPLE OF ELEMENTARY NEUTRALIZATION LOGBOOK

Katahdin Analytical Services, Inc. – Elementary Neutralization Logbook

Date: 5-9-13		Time: 16:30	Analyst: GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
6	7	good	
6	7		
5	6		
5	5		
5	7		

Date: 5-16-13		Time: 12:00	Analyst: GN/WS
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
5	7	good	
5	5		
5	7		
5	7		
6	5		
6	8		
5	7		
5	7		
4	6		

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Galen Nickerson

Review Date: 12-8-14

SOP Number: SD-903

SOP Title: Sample Disposal

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: Deborah J. Nadeau **Date:** 12.8.14

QAO Signature: Jessie Dimond **Date:** 12.08.14

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP:

Review Date: 02/02/16

SOP Number: SD-903-05

SOP Title: Sample Disposal

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 2-2-16

QAO Signature: Jessie Dimond **Date:** 02.03.16

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP:

Review Date: 02/02/16

SOP Number: SD-903-05

SOP Title: Sample Disposal

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 2-2-16

QAO Signature: Leslie Dimond **Date:** 02.03.16

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Gale Nickerson

Review Date: 5-13-16

SOP Number: SD-903-05

SOP Title: Sample Disposal

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: Gale Nickerson **Date:** 05-13-16

QAO Signature: Lucio Dimond **Date:** 05.13.16

change waste check list figure: Daily → weekly

Title: Perfluorinated Compounds (PFCs) in Water, Soils, Sediments and Tissue
[Method 537 Modified]

Approvals (Signature/Date):

Robert Hrabak 12/8/16 Date Technical Manager	Joe Schairer 12/9/16 Date Health & Safety Manager / Coordinator
Lisa Stafford 12/9/16 Date Quality Assurance Manager	Crystal Pollock for 12/9/16 Date Laboratory Manager

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water, soil, sediment and tissue samples for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS).

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
Perfluoro-n-hexanoic acid	PFHxA	307-24-4
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluoro-n-decanoic acid	PFDA	335-76-2
Perfluoro-n-undecanoic acid	PFUdA	2058-94-8
Perfluoro-n-dodecanoic acid	PFDoA	307-55-1
Perfluoro-n-tridecanoic acid	PFTrDA	72629-94-8
Perfluoro-n-tetradecanoic acid	PFTeDA	376-06-7
Perfluoro-n-hexadecanoic acid	PFHxDA	67905-19-5
Perfluoro-n-octadecanoic acid	PFODA	16517-11-6
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
Perfluoro-1-heptanesulfonic acid	PFHpS	375-92-8
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1
Perfluoro-1-decanesulfonic acid	PFDS	335-77-3
Perfluorinated sulfonamides (FOSA)		
Perfluoro-1-octanesulfonamide	FOSA	754-91-6
N-ethylperfluoro-1-octanesulfonamide	EtFOSA	4151-50-2
N-methylperfluoro-1-octanesulfonamide	MeFOSA	31506-32-8
Perfluorinated sulfonamidoacetic acids (FOSAA)		
N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2991-50-6
N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA	2355-31-9
Fluorotelomer sulfonates (FTS)		
1H,1H,2H,2H-perfluorooctane sulfonate *(6:2)*	6:2 FTS	27619-97-2
1H,1H,2H,2H-perfluorodecane sulfonate *(8:2)*	8:2 FTS	39108-34-4

Sample results for PFOA may also be reported as APFO, at the request of the client. (See Section 12.7)

1.2. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	250 mL	2.0 ng/L – 100 ug/L	2 ng/L to 400 ug/L
Soil/Sediment/Tissue	5 g	0.2 ug/kg – 20 ug/kg	0.2 to 100 ug/kg

1.3. Due to poor chromatographic peak shape which degraded with repeated injections for Perfluoro-1-octanesulfonamidoamide (FOSSA), this analyte is no longer included in the method.

1.4. The procedure for the analysis of water samples via direct aqueous injection (DAI) for a subset of the list in Section 1.1 using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a SCIEX 5500 is described in the Appendix to this SOP.

1.5. When undertaking projects for Department of Defense (DoD) and/or Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, "Federal Program Requirements" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Water samples are extracted using a solid phase extraction (SPE) cartridge, unless EtFOSA and MeFOSA are requested. PFCs are eluted from the cartridge with ammonium hydroxide/methanol solution.

2.2. Soil samples are extracted with KOH/methanol solution using an orbital shaker for 3 hours followed by sonication for 12 hours. The mixture is centrifuged and the solvent filtered.

2.2.1. Optional cleanups may include sample freezing and/or cleanup by SPE cartridge, unless EtFOSA and MeFOSA are requested.

2.3. The final 80:20 methanol:water extracts are analyzed by LC/MS/MS. PFCs are separated from other components on a C18 column with a solvent gradient program using 20 mM Ammonium Acetate/water and methanol. The mass spectrometer detector is operated in the electrospray (ESI) negative ion mode for the analysis of PFC.

2.4. Isotope dilution technique is employed with this method for most compounds of interest. The isotope dilution analytes (IDA's) consist of carbon-13 labeled analogs, oxygen-18 labeled analogs or deuterated analogs of the compounds of interest, and they are spiked into the samples at the time of extraction. This technique allows correction for analytical bias encountered when analyzing more chemically complex

environmental samples, because the isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have a labeled analog are quantitated by IDA method using a closely related labeled analog.

2.5. Quantitation by the external standard method is employed for the IDA analytes and assumes a proportional relationship between the initial calibration and the analyte in the extract. The ratio of the peak response to mass or concentration injected is used to prepare a calibration curve. Peak response is measured as the area of the peak. Isotope dilution technique is employed with this method for most compounds of interest. The IDA's consist of carbon-13 labeled analogs or oxygen-18 labeled analogs of the compounds of interest, and they are spiked to the samples at the time of extraction. This technique allows correction for analytical bias encountered when analyzing more chemically complex environmental samples, because the isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have a labeled analog are quantitated by IDA method using a closely related labeled analog.

3. DEFINITIONS

3.1. PFCAs: Perfluorocarboxylic acids

3.2. PFSAs: Perfluorinated sulfonates

3.3. FOSA: Perfluorinated sulfonamides

3.4. PFOA: Perfluorooctanoic acid (may also be written PHOA).

3.5. APFO: Ammonium perfluorooctanoate

3.6. PFOS: Perfluorooctane sulfonate (may also be written PHOS)

3.7. MPFOA: Perfluoro-n-[1,2,3,4-13C4]octanoic acid. Carbon-13 labeled PFOA

3.8. MPFOS: Perfluoro-1-[1,2,3,4-13C4]octanesulfonate. Carbon-13 labeled PFOS

3.9. PTFE: Polytetrafluoroethylene (e.g., Teflon®)

3.10. SPE: Solid phase extraction.

3.11. PP: Polypropylene

3.12. PE: Polyethylene

3.13. HDPE: High density polyethylene

3.14. IDA: Isotope dilution analytes

3.15. Further definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

4. INTERFERENCES

4.1. PFCs have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed below in Section 6.

4.2. To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.

4.3. PTFE products can be a source of PFOA contamination. The use of PTFE in the procedure should be avoided or at least thoroughly tested before use. Polypropylene (PP) products may be used in place of PTFE products to minimize PFOA contamination.

4.3.1. Standards and samples are injected from polypropylene autosampler vials with polypropylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.

4.3.2. Random evaporation losses have been observed with the polypropylene caps causing high IDA recovery after the vial was punctured and sample re-injected. For this reason, it is best to inject standards and samples once in the analytical sequence.

4.3.3. Teflon-lined screw caps have detected PFCs at low concentrations. Repeated injection from the same teflon-lined screw cap have detected PFNA at increasing concentration as each repeated injection was performed, therefore, it is best to use polypropylene screw caps.

4.4. Volumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFOA. These items should be labeled for use only with similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.

4.5. Commercial sources of PFOS may produce several peaks in the PFOS chromatogram. These adjacent peaks are either completely resolved or not resolved but with a profound deflection that can be resolved during peak integration. The later of the

peaks matches the retention time of the single labeled PFOS peak. Earlier peaks are branched isomers of PFOS, rather than a result of peak splitting. The earlier peak is included during peak integration.

4.6. The phenomenon of the linear and branched isomers of PFOS exists for other PFAS, such as PFHxS and PFBS. Thus, in an attempt to reduce PFOS bias, it is required that *m/z* 449>80 transition be used as the quantitation transition.

4.7. Both branched and linear PFAAs can potentially be found in the environment. For the compounds that give rise to more than one peak, all the chromatographic peaks observed in the standard and/or sample must be integrated and the areas included.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns

5.1.1. Preliminary toxicity studies indicate that PFCs could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFCs must be handled in the laboratory as hazardous and toxic chemicals.

5.1.2. Exercise caution when using syringes with attached filter disc assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.1.3. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.4. Eye protection that satisfies ANSI Z87.1 (as per the TestAmerica Corporate Safety Manual), laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.1.5. Perfluorocarboxylic acids are acids and are not compatible with strong bases.

5.1.6. The use of vacuum systems presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.7. Glass containers are not to be used for "tumbling" soil samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material[1]	**Hazards**	**Exposure Limit[2]**	**Signs and Symptoms of Exposure**
Acetic Acid (3-2-1)	Corrosive Poison Flammable	10 ppm-TWA 15 ppm-STEL	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage to the upper respiratory tract. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent damage, including blindness. Brief exposure to 5000 PPM can be fatal.
Hexane (2-3-0)	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Can cause pain and severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause deep ulcerations to skin, permanent eye damage, circulatory failure and swallowing may be fatal.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Potassium Hydroxide	Corrosive Poison		Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
Sodium Hydroxide	Corrosive Poison	2 mg/cm^3 (Ceiling)	Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6. EQUIPMENT AND SUPPLIES

6.1. 8 mL test tubes, screw thread, with caps.

6.2. 15 mL polypropylene test tubes with polypropylene screw caps.

6.3. 50 mL graduated plastic centrifuge tubes.

6.4. 125 mL PPE wide-mouth bottles.

6.5. 16 oz or 500 mL PPE bottles with PPE screw caps.

6.6. Analytical balance capable of accurately weighing to the nearest 0.0001g, and checked for accuracy each day it is used in accordance with WS-QA-0041.

6.7. Syringe filter, Millipore Millex-HV 0.45 um, or equivalent. Do not use PTFE type filters.

6.8. 300-µL autosampler vials, polypropylene, with polypropylene screw caps, Waters PN 1860004112, or equivalent.

6.9. SPE columns

6.9.1. Phenomenex Strata SPE C18, 6 mL, 500 mg, part number 8B-S002-HCH, Waters SepPak C18, 1 to 10g, or equivalent.

6.9.2. Waters Oasis WAX 150 mg/6 cc (PN 186002493) for the cleanup of solids.

6.9.3. Waters Oasis WAX 500 mg/6 cc (PN 186004647) for extraction of PFC from aqueous sample.

6.9.4. Phenomonex Gemini 3 µm C18 110Å, 50 X 2 mm, Part No. 00B-4439-B0.

6.9.5. Phenomonex Luna 5 µm C18(2) 100Å, 30 X 3 mm, Part No. 00A-4252-Y0.

6.10. PFC Isolator column, Waters PN 186004476, plumbed between the UPLC pumps and autosampler valve to minimize PFC background from the UPLC solvent lines and filters.

6.11. Granulated carbon.

6.12. Vacuum manifold for Solid Phase Extraction (SPE).

6.13. Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc.). These should be disposable where possible, or marked and segregated for high-level versus low-level use.

6.14. WATERS Acquity UPLC system connected to Triple Quad MS such as Waters Micromass Quattro Premier XE or SOILEX 5500 Triple Quad MS. These systems utilize Chrom Peak Review, version 2.1 or equivalent.

6.15. Acquity UPLC BEH C18 1.7 um, 3.0 mm x 150 mm, Part No. 186004690, Phenomenex Gemini-NX C18 3 um, 3.0 mm x 100 mm, Part No. 00D-4453-Y0, Shimadzu CTO-20AC HPLC equipped with 3 LC-20D pumps and one DGU-20 degassing unit or equivalent.

6.16. Bottle rotating apparatus for soil extractions.

6.17. Glass fiber filter, Whatman GF/F, catalog number 1825 090 or equivalent.

6.18. Preventive and routine maintenance is described in the table below

HPLC/MS/MS Preventative Maintenance
As Needed:
Change pump seals.
Change in-line filters in autosampler (HPLC).
Check/replace in-line frit if excessive pressure or poor performance.
Replace column if no change following in-line frit change.
Clean corona needle.
Replace sample inlet tube in APCI (10.1 cm).
Replace fused silica tube in ESI interface.
Clean lenses.
Clean skimmer.
Ballast rough pump 30 minutes.
Daily (When in use)
Check solvent reservoirs for sufficient level of solvent.
Verify that pump is primed, operating pulse free.
Check needle wash reservoir for sufficient solvent.
Verify capillary heater temperature functioning.
Verify vaporizer heater temperature.
Verify rough pump oil levels.
Verify turbo-pump functioning.
Verify nitrogen pressure for auxiliary and sheath gasses.
Verify that corona and multiplier are functioning.
Semi-Annually
Replace rough-pump oil (4-6 months).
Replace oil mist and odor elements.
Replace activated alumina filter if applicable.
Annually
Vacuum system components including fans and fan covers.
Clean/replace fan filters, if applicable.

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1. Acetic Acid, glacial

7.1.2. Ammonium acetate (20 mM in water)

7.1.3. Ammonium Hydroxide (NH4OH), 0.3% in methanol

7.1.4. Hexane

7.1.5. Hydrochloric Acid (HCl), 2.0 M solution in water

7.1.6. Methanol

7.1.7. Potassium Hydroxide (KOH), 0.4% in methanol

7.1.8. Ottawa Sand

7.1.9. Sodium Hydroxide (NaOH), 0.1N, in water

7.1.10. Water, Nanopure or Millipore, must be free of interference and target analytes

7.2. Standards

7.2.1. PFCs are purchased as a high purity solids (96% or greater) or as certified concentration in solution. Standard materials are verified compared to a second source material at the time of initial calibration. The solid stock material is stored at room temperature or as specified by manufacturer or vendor.

7.2.2. If solid material is used for preparing a standard, stock standard solutions are prepared from the solids and are stored at 4 ± 2°C. Stock standard solutions should be brought to room temperature before using. Standards are monitored for signs of degradation or evaporation. Standard solutions must be replaced at least annually from the date of preparation.

7.2.3. PFBS, PFHxS, PFHpS, PFOS, PFDS, MPFOS, and many other PFASs are not available as the acids, but rather as their corresponding salts, such as sodium or potassium. The standards are prepared and corrected for their salt content according to the equation below.

7.2.4. $Mass_{acid}$ = Measured $Mass_{salt} \times MW_{acid} / MW_{salt}$

7.2.5. Where: MW_{acid} is the molecular weight of PFAA

7.2.6. MW_{salt} is the molecular weight of the purchased salt.

7.2.7. For example, the molecular weight of PFOS is 500.1295 and the molecular weight of NaPFOS is 523.1193. Therefore, the amount of NaPFOS used must be adjusted by a factor of 1.046

7.3. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of PFOA and PFOS stock solutions in 80% methanol/water. The calibration stock solution is

diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.4. Initial Calibration (ICAL) Levels (ng/mL)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
Perfluoroalkylcarboxylic acids (PFCAs)							
PFBA	0.5	1.0	5.0	20	50	200	400
PFPeA	0.5	1.0	5.0	20	50	200	400
PFHxA	0.5	1.0	5.0	20	50	200	400
PFHpA	0.5	1.0	5.0	20	50	200	400
PFOA *	0.5	1.0	5.0	20	50	200	400
PFNA	0.5	1.0	5.0	20	50	200	400
PFDA	0.5	1.0	5.0	20	50	200	400
PFUdA	0.5	1.0	5.0	20	50	200	400
PFDoA	0.5	1.0	5.0	20	50	200	400
PFTrDA	0.5	1.0	5.0	20	50	200	400
PFTeDA	0.5	1.0	5.0	20	50	200	400
PFHxDA	0.5	1.0	5.0	20	50	200	400
PFODA	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonic acids (PFSAs)							
PFBS	0.5	1.0	5.0	20	50	200	400
PFHxS *	0.5	1.0	5.0	20	50	200	400
PFHpS	0.5	1.0	5.0	20	50	200	400
PFOS	0.5	1.0	5.0	20	50	200	400
PFDS	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonamides (FOSA)							
FOSA	0.5	1.0	5.0	20	50	200	400
EtFOSA	0.5	1.0	5.0	20	50	200	400
MeFOSA	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonamidoacetic acids (FOSAA)							
EtFOSAA	0.5	1.0	5.0	20	50	200	400
MeFOSAA	0.5	1.0	5.0	20	50	200	400
Fluorotelomer sulfonates (FTS)							
6:2 FTS	0.5	1.0	5.0	20	50	200	400
8:2 FTS	0.5	1.0	5.0	20	50	200	400
Labeled Isotope Dilution Analytes (IDA)							
MPFBA	50	50	50	50	50	50	50
M5PFPeA	50	50	50	50	50	50	50
MPFHxA	50	50	50	50	50	50	50
MPFHpA	50	50	50	50	50	50	50

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
M4PFOA	50	50	50	50	50	50	50
MPFNA	50	50	50	50	50	50	50
MPFDA	50	50	50	50	50	50	50
MPFUdA	50	50	50	50	50	50	50
MPFDoA	50	50	50	50	50	50	50
MPFHxS	50	50	50	50	50	50	50
MPFOS	50	50	50	50	50	50	50
M8FOSA	50	50	50	50	50	50	50
D5-EtFOSA	50	50	50	50	50	50	50
D3-MeFOSA	50	50	50	50	50	50	50
D5-EtFOSAA	50	50	50	50	50	50	50
D3-MeFOSAA	50	50	50	50	50	50	50
M2-6:2FTS	50	50	50	50	50	50	50
M2-8:2FTS	50	50	50	50	50	50	50

* both branched and linear isomers are used.

Note: Sample extracts are in 80% MeOH/H_2O.

FOSAA may be added to the mix and are added at the same concentration as FOSA.

Note- The above calibration limits are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.

7.4.1. A technical (qualitative) grade PFOA standard is analyzed initially, then after ICAL when a new column is installed or when significant changes are made to the HPLC parameters. This solution is used as a reference for the PFOA isomers (branched and linear) retention times.

7.5. Initial Calibration Verification Standard (ICV).

A second source solution for PFC is purchased from the same vendor; the PFC-MXB contains most of the target analytes in this mixture and is used as an ICV. A few compounds are not available in this mixture, may not be available as another lot, and are not available from another vendor. For these analytes only, a second analyst may prepare a second source standard from the same source as the ICAL to produce an ICV. The recommended concentration of the ICV standard should be in the mid-range of the calibration curve. The concentration may be adjusted if the initial calibration levels are changed or altered. The IS is added at a fixed concentration of 50 ng/mL.

7.6. LCS/Matrix PFC Spike Solution, 500 ng/mL.

The PFC spike solution is prepared by diluting all PFCs to produce a solution containing PFCs each at 500 ng/mL in methanol.

7.7. PFC Isotope Dilution Analyte Solution, 1000 ng/mL.

The PFC-IS solution is prepared by diluting all labeled PFCs to produce a solution each at 1000 ng/mL in methanol.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Soil samples are collected in pre-cleaned 250 mL HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6°C for shipment to the laboratory.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6°C. Water samples must be extracted within 14 days of collection. Soil samples must also be extracted within 14 days of collection. Tissue samples must be extracted within 1 year of collection if stored at -20°C. Extracts must be refrigerated at 0 - 6°C, and analyzed within 40 days from extraction.

NOTE: As of this writing, Method 537 provides for a 14 day holding time for water samples preserved with Trizma buffer. The scientific literature indicates that perfluorinated substances are highly persistent in the environment. TestAmerica Sacramento has conducted holding time studies that support a 14 day holding time for aqueous samples with and without Trizma preservation. TestAmerica Denver has conducted stability studies indicating that medium- and low-level solutions of PFOA are stable for at least three months in polystyrene and polypropylene plastics at 0-6C. The 14 days/40 day holding times given above are based on the holding time study and general EPA convention for the holding time of extractable organic compounds in water and soil.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability (IDOC)

The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.2. Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC program document (WS-PQA-003) for further details of the batch definition.

9.2.1. The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a matrix spike/matrix spike duplicate (MS/MSD), a laboratory control sample (LCS) and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count toward the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be

replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD, an LCSD may be substituted if batch precision is required by the program or client. In the event that multiple MS/MSDs are run with a batch due to client requirements, the additional MS/MSDs do not count toward the maximum 20 samples in a batch.

9.3. One method blank (MB, laboratory reagent blank) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. For solid sample, the method blank is an aliquot of Ottawa sand. The method blank is processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside of the control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria.

9.3.1. If the MB produces a peak within the retention time window of any of the analytes determine the source of the contamination and eliminate the interference before processing samples.

9.3.2. The method blank must not contain any analyte at or above the reporting limit, or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.3.3. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.4. Re-extraction and re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.3.5. Refer to WS-PQA-003 for further details of the corrective actions.

9.3.6. Projects performed under the auspices of the DOD/DOE must meet QSM specific criteria for method blanks. Results are acceptable if the blank contamination is less than ½ of the reporting limit for each analyte, or less than 1/10 of the regulatory limit, or less than 1/10 of the sample result for the same analyte, whichever is greater. If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. If contamination remains, the contaminated samples should be re-prepared and reanalyzed with a new MB and batch-specific QC samples.

9.4. A laboratory control sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water for aqueous samples and Ottawa sand for solids) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside of the control limits. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See WS-PQA-0003 for specific acceptance criteria. The control limits for the LCS are stored in TALS.

9.5. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside of the control limits must be within the control limits in the LCS. Corrective actions must be documented on a nonconformance memo, then implemented when recoveries of any spiked analyte are outside of the control limits provided by the LIMS or by the client.

9.6. A duplicate control sample (LCSD or DCS) may be added when insufficient sample volume is provided to process an MS/MSD pair, or is requested by the client. The LCSD is evaluated in the same manner as the LCS. See WS-PQA-003 for specific acceptance criteria.

9.7. Initial calibration verification (ICV) – When available, a second source standard is analyzed with the initial calibration curve. The concentration should be at the mid range of the curve.

Corrective actions for the ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards.

9.8. Isotope Dilution Analytes

9.8.1. The IDA solution is added to each field and QC sample at the time of extraction, as described in Section 11. As described in Section 7, this solution consists of isotopically labeled analogs of the analytes of interest.

9.8.2. IDA recoveries are flagged if they are outside of the acceptance limits (25–150%). Quantitation by isotope dilution generally precludes any adverse

effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-nose ratio is greater than 10:1.

9.8.2.1. Evaluate data quality for usability, flag and submit a non-conformance memo for any analytes outside of the recovery criteria, and report if data is deemed not adversely effected.

9.8.2.2. Re-extraction of samples should be performed if the signal-to-noise for any IDA is less than 10:1 or if the IDA recoveries fall below 10%.

9.8.2.2.1. Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-P-003 “Calibration Curves and Selection of Calibration Points”.

10.2. Routine instrument operating conditions are listed in the table in Section 11.6.1.

10.3. Instrument Tuning

Instrument tuning is done initially when the method is first developed and thereafter as needed to maintain the sensitivity and selectivity of the method. Tuning is done by infusing each individual compound (native, IDA and recovery) into the mobile phase using a tee fitting at a point just before the entrance to the electrospray probe. The responses for the parent and daughter ions for each compound are observed and optimized for sensitivity and resolution. Mass assignments are reviewed and calibrated if necessary. The mass assignments must be within ± 0.5 amu of the values shown in the table in Section 11.5.1.

10.4. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include, but are not limited to new columns or pump seals. A new calibration is not required after minor maintenance.

10.5. With the exception of the circumstances delineated in policy P-T-001, it is not acceptable to remove points from a calibration curve. In any event, at least five points must be included in the calibration curve. Average Response Factor and linear fit calibrations require five points, whereas Quadratic (second order) calibrations require six points.

10.6. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.

10.7. All units used in the calculations must be consistently uniform, such as concentration in ng/mL.

10.8. Initial Calibration

10.8.1. A number of analytical standards of different analyte concentrations are used to generate the curve. Each standard is injected once to obtain the peak response for each analyte at each concentration. These standards define the working range of the analysis.

10.8.1.1. A minimum of five analytical standards is used when using average response factor and or linear calibration fits.

10.8.1.2. A minimum of six analytical standards is used for quadratic fit to generate the curve.

10.8.2. Calibration is by average response factor, linear fit, or by quadratic fit. Quadratic fit is used for the analyte if the response is non-linear.

10.8.2.1. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated by isotope dilution must be < 35% for the curve to be valid.

10.8.2.2. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated by IDA must be < 50% for the curve to be valid.

10.8.2.3. For linear fit, the intercept of the line must be less than ½ the reporting limit, and the coefficient of determination (r2) must be greater than or equal to 0.990 for the curve to be considered valid (or the correlation coefficient (r) > 0.995).

10.9. Calibration Curve Fits

10.9.1. Linear regression or quadratic curves may be used to fit the data to a calibration function. Detailed descriptions and formulas for each fitting type can be found in SOP CA-Q-S-005, “Calibration Curves (General)”.-

10.9.2. The linear curve uses the following function:

Equation 1

$$y = bx + c$$

Where:

$$y = \frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$$

x = concentration
b = slope
c = intercept

10.9.3. The quadratic curve uses the following function:

Equation 2

$$y = ax^2 + bx + c$$

Where y, x, b, and c are the same as above, and a = curvature.

10.9.4. The external standard method uses the following equation:

Equation 3

$$\text{Response Factor} = \frac{\text{Peak Area}}{\text{Concentration of Solution } (ng/mL)}$$

10.9.5. Evaluation of Calibration Curves

The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- The absolute value of the intercept of a regression line (linear or non-linear) at zero response must be less than the reporting limit.
- There should be no carryover at or above 1/2 MRL after a high CAL standard.

If these criteria are not met, instrument conditions and standards will be checked, and the ICAL successfully repeated before continuing.

10.9.6. Weighting of Calibration Points

In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. Because accuracy at the low end of the curve is very important for this analysis, it is preferable to increase the weighting of the lower concentration points. 1/concentration or 1/x weighting is encouraged. Visual inspection of the line fitted to the data is important in selecting the best fit.

10.10. Initial Calibration Blank (ICB)

10.10.1. Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of 80:20 methanol:water blank.

10.10.2. The result for the calibration blank must be less than the reporting limit.

10.10.3. If the ICB is greater than the reporting limit then the source of contamination

must be identified and any necessary cleaning completed, and then the instrument should be recalibrated.

10.11. Initial Calibration Verification (ICV)

10.11.1. Following the ICAL and the ICB, an ICV standard obtained from a different source or vendor than the ICAL standards is analyzed. This ICV standard is a mid-range standard.

10.11.2. The recovery for the ICV must meet the appropriate following criteria;

10.11.2.1. The native analyte must be within or equal to 60-140% for all natives quantitated by isotope dilution.

10.11.2.2. The native must be within or equal to 50-150% for all natives quantitated by IDA.

10.11.2.3. The IDA must be $\geq$ 50 and $\leq$ 150%.

10.11.3. See Section 9.7 for corrective actions in the event that the ICV does not meet the criteria above.

10.12. Continuing Calibration Verification (CCV) – At the beginning of a run, the end of a run, and after every 10 samples are analyzed a CCV must be injected to determine if the calibration is still valid. The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required. The CCVs are usually at the mid level range of the curve and should vary throughout the run. The curve and ICV do not need to be run every day. To start an analytical run a CCV can be analyzed and if it meets acceptance criteria a run can be started. In addition the low standard in the curve must be analyzed and must be within ± 50% of the expected value.

10.12.1. The recovery for the CCV standards must be equal to or within 60-140% for all natives quantitated by isotope dilution and equal to or within 50% to 150% for all natives quantitated by IDA. The recovery for the IDA $\geq$50 to $\leq$150%.

10.12.2. If this is not achieved, the instrument has drifted outside the calibration limits. The instrument must be recalibrated.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an Non-Conformance Memo (NCM). The NCM process is described in more detail in SOP WS-QA-0023. The NCM shall be filed in the project file and addressed in the case narrative.

11.2. Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3. Water Sample Preparation

11.3.1. Visually inspect samples for the presence of settled and or suspended sediment. If sediment is apparent, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration.

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.3.2. Measure 250 mL of each sample using a graduated cylinder and pour into a labeled 16 oz polypropylene (PP) bottle. *Prepare separate aliquots of 1.0 mL if EtFOSA and/or MeFOSA are requested.)*

11.3.3. Prepare additional aliquots of a field sample for the MS/MSD, if requested.

11.3.4. Prepare two 250 mL aliquots of HPLC-grade water for the method blank and LCS. (or 1.0 mL if EtFOSA and/or MeFOSA are requested.)

11.3.5. Spike the LCS and MS/MSD (if requested) with 0.020 mL (20 uL) of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 40 ng/L. If EtFOSA and/or MeFOSA are required, increase the amount of LCS Matrix PFC spike solution added to 200 uL.

11.3.6. Add 0.025 mL (25 uL) of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial. If EtFOSA and/or MeFOSA are requested increase the amount of IDA added to 125 uL.

11.3.7. If EtFOSA and/or MeFOSA are requested adjust the final volume (FV) of these aliquots to 5.0 mL with MeOH. Vortex each sample. Then transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.4. **Solid Phase Extraction (SPE)** – (Do not perform SPE clean up if EtFOSA and/or MeFOSA are requested.)

The automated Zymark Auto-Trace Workstation can be used as long as the program follows these conditions and passes the PFC background check.

11.4.1. Condition the SPE cartridges (Waters WAX, 500 mg/6 cc) by passing the

following without drying the column.

NOTE: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.4.2. Wash with 5.0 mL of 0.3% NH4OH/methanol.

11.4.3. Wash with 5.0 mL of 0.1N NaOH/water. Close valve when ~ 200 uL remains on top to keep column wet. After this step, the columns cannot go dry until the completion of loading and rinsing samples.

11.4.4. Appropriately label the columns and add the reservoir to the column.

11.4.5. Add samples to the columns and with vacuum, pull the entire 250 mL aliquot of the sample through the cartridge at rate of approximately 2 to 5 drops per second.

11.4.6. After the final loading of the sample but before completely passed through the column, rinse the SPE column with 1 mL of water.

11.4.7. After the sample and water rinse has completely passed through the cartridge, allow the column to dry well with vacuum for 15 minutes.

11.5. SPE Column Wash of Aqueous with hexane –

11.5.1. Load the first 5 mL of hexane to soak for five minutes, then elute to waste.

11.5.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

11.5.3. Allow the column to dry with vacuum for 5 to 10 minutes. Columns must be dried before continuing.

11.6. **SPE Elution** – using 15 mL polypropylene test tube as receiving tube in the SPE manifold.

11.6.1. Rinse samples bottles with 5 mL of 0.3% NH_4OH/methanol and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.6.2. Repeat sample bottle to column reservoir rinse and cartridge elution with a

second 5 mL aliquot of 0.3% NH_4OH/methanol. The total collection should be approximately 10 mL.

11.7. Extract Concentration

11.7.1. Concentrate each sample under a gentle stream of nitrogen to near dryness.

11.7.2. Add 400 uL of methanol to each extract, soak and vortex to mix well to reconstitute extract.

11.7.3. Add 100 uL of water to each sample for a final solvent composition of 80:20 Methanol:Water and vortex to mix the mixture well.

11.7.4. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.7.5. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFCs.

11.8. Soil, Sediment and Tissue Sample Preparation and Extraction

11.8.1. Visually inspect soil samples for homogeneity.

11.8.2. Weigh a representative 5 g aliquot of soil, sediment or tissue sample into a 50 mL polypropylene wide-mouth bottle. Weigh additional sample amounts for the matrix spike and matrix spike duplicate analyses if they are requested. *(Prepare separate aliquots if EtFOSA and/or MeFOSA are requested.)*

11.8.3. For the method blank and LCS matrix, use 5 g each of Ottawa sand.

11.8.4. Spike the LCS and MS/MSD (if requested) with 0.040 mL (40 uL) of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 4.0 ng/g.

11.8.5. Add 0.05 mL (50 uL) of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.

11.8.6. Cap the bottles and allow the spike to settle into the sample matrix. Gently shake the bottles to mix the spike into the matrix.

11.8.7. Add 20 mL of 0.4% KOH/methanol to each sample.

11.8.8. Shake each sample on an orbital shaker at room temperature for 3 hours.

11.8.9. Following the shaking, extract the samples in an ultrasonic water bath for an

additional 12 hours.

11.8.10. After the completion of extraction, centrifuge each sample at 3500 rpm for 15 minutes.

11.8.11. Collect and decant the KOH/methanol extract to a new 50-mL centrifuge tube.

11.8.12. Add another 2 mL of 0.4% KOH/methanol solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.

11.8.13. Combine the rinsate to the first corresponding tubes.

11.8.14. To the final KOH/methanol extract, add 2 mL of water to each. *(Omit this step if EtFOSA and/or MeFOSA are requested.)*

11.8.15. Concentrate the KOH/methanol/water extract under nitrogen to less than 2 mL, and dilute with water to 15 mL final volume. *(Omit this step if EtFOSA and/or MeFOSA are requested.)*

11.8.16. Acidify with 80 uL of glacial acetic acid, and mix the contents well with vortex mixer. Check the pH to ensure pH is between 6 to 8.

11.8.17. Centrifuge at 3500 rpm for 15 minutes.

11.9. **Solid Cleanup by SPE.** (Do not perform SPE clean up if EtFOSA and/or MeFOSA are requested. Proceed directly to Section 11.12)

11.9.1. Set up WAX 150 mg/6 cc SPE columns for sample cleanup using vacuum manifold.

11.9.2. Condition the SPE cartridges by passing the following without drying the column.

NOTE: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.9.3. Wash with 5.0 mL of 0.3% NH4OH/methanol.

11.9.4. Wash with 10 mL of 0.1N NaOH/water. Close valve when ~ 500uL remains on top of column to keep column wet. *After this step, the columns cannot go dry until the completion of loading and rinsing samples.*

11.9.5. Add extracts to the columns and with vacuum, pull the entire extracts through the cartridge at rate of approximately 3 to 5 drops per second.

11.9.6. Rinse the sample tube with 5 mL of water and add to the SPE column.

11.9.7. Dry the columns with vacuum for 15 minutes.

11.10. SPE Column Wash of solids with hexane –

11.10.1. Load the first 5 mL of hexane to soak for five minutes, and elute to waste.

11.10.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

11.10.3. Allow the column to dry with vacuum for 10 minutes. Columns must be dried before continuing.

11.11. **SPE Elution** – using 15 mL polypropylene test tube as receiving tube in the SPE manifold.

11.11.1. Elute the analytes from the cartridge with 5.0 mL of 0.3% NH4OH/methanol, first allow the solution to soak for 5 minutes, and then elute into the 15 mL collection tube.

11.11.2. Add a second 5 mL of 0.3% NH4OH/methanol and collect the eluant into the collection tube. The total collection should be approximately 10 mL.

11.12. Extract Concentration

11.12.1. Concentrate each sample under a gentle stream of nitrogen to near dryness.

11.12.2. Add 800 uL of methanol to each extract, soak and vortex to mix well to reconstitute extract.

11.12.3. Add 200 uL of water to each sample for a final solvent composition of 80:20 Methanol:Water and vortex to mix the mixture well.

11.12.4. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.12.5. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFCs.

11.13. Other types of Sample Cleanup

11.13.1. **Freezing technique** to remove lipids.

11.13.1.1. If samples contain lipids then freeze the methanolic extract and QC extracts at -20°C for at least 1 hour. Collect the solvent layer.

11.13.2. **Cleanup with graphitized carbon** which may also be used to remove organic interferences.

11.13.2.1. Add 100 mg of graphitized carbon to each sample extract and QC extracts.

11.13.2.2. Shake vigorously and then let sit for 10 minutes.

11.13.2.3. Centrifuge each sample for 2 minutes at 1000 rpm.

11.13.2.4. Decant the solvent layer

11.13.3. Concentrate each sample under a gentle stream of nitrogen to approximately 0.5 mL.

11.13.4. Add 200 uL of Millipore water to each sample.

11.13.5. Bring the final volume to 1.0 mL with methanol (80% methanol/20% water).

11.13.6. Filter through a 0.45 µm syringe filter as necessary or centrifuge the extracts to obtain a clear supernant. *Note: Syringe filter should be checked for PFC background before using.*

WARNING: Application of excessive pressure has caused disc filters to rupture and burst. Exercise discretion when filtering.

11.14. Instrument Analysis

11.14.1. Suggested operation conditions are listed below:

Waters Acquity UPLC

Routine Instrument Operating Conditions					
HPLC Conditions (Waters Acquity UPLC)					
Column (Column temp = 50°C)	Waters Acquity BEH 1.7µm C18, 3.0 x 150 mm				
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol				
Gradient Program	**Time**	**%A**	**%B**	**Curve**	**Flow Rate mL/min.**
	0	98	2	6	0.30
	1	98	2	6	0.30
	2	50	50	6	0.30

	12	10	90	6	0.30
	12.5	0	100	6	0.30
	16	0	100	6	0.30
	16.2	98	2	6	0.30
	Maximum Pressure limit = 15,000 psi				
Injection Size	10 µL (fixed amount throughout the sequence)				
Run Time	~20 minutes				

Mass Spectrometer Interface Settings (Quattro Premier XE)	
MS Interface Mode	ESI Negative Ion
Capillary (kV)	2.8
Cone (V)	Varies from 8.0 to 65
Extractor (V)	3
Source Temp	135ºC
Desolvation Temp	350ºC
Cone Gas (nitrogen) Flow	25 L/hour
Desolvation Gas (nitrogen) Flow	1100 L/hour

Mass Spectrometer Scan Settings						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
PFBA	Perfluorobutanoic acid	213 > 169	0.02	8	10	1
13C4-PFBA	IDA	217 > 172	0.02	12	10	1
PFPeA	Perfluoropentanoic acid	263 > 219	0.02	10	10	2
13C5PFPeA	IDA	268 > 223	0.02	11	9	2
PFBS	PerfluorobutanesulfonIc acid	299 > 80	0.02	45	35	2
PFHxA	Perfluorohexanoic acid	313 > 269	0.02	10	10	3
13C2-PFHxA	IDA	315 > 270	0.02	12	9	3
PFHpA	Perfluoroheptanoic acid	363 > 319	0.02	10	10	4
13C4-PFHpA	IDA	367 > 322	0.02	12	10	4
PFHxS	Perfluorohexanesulfonic acid	399 > 80	0.02	55	35	4
18O2-PFHxS	IDA	403 > 84	0.02	50	40	4
PFOA	Perfluorooctanoic acid	413 > 369	0.02	12	10	5
13C4PFOA	IDA	417 > 372	0.02	12	12	5
PFHpS	Perfluoroheptanesulfonate	449 > 80	0.02	60	38	5
PFNA	Perfluorononanoic acid	463 > 419	0.02	16	10	7
13C5-PFNA	IDA	468 > 423	0.02	12	12	7
PFOS	Perfluorooctanesulfonic acid	499 > 80	0.02	60	40	6
13C4-PFOS	IDA	503 > 80	0.02	35	48	6
PFDA	Perfluorodecanoic acid	513 > 469	0.02	16	12	8
813C2-PFDA	IDA	515 > 470	0.02	14	12	8
PFUdA	Perfluoroundecanoic acid	563 > 519	0.02	15	12	10

Mass Spectrometer Scan Settings						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
13C2-PFUdA	IDA	565 > 520	0.02	14	12	10
PFDS	Perfluorodecanesulfonic acid	599 > 80	0.02	74	48	10
FOSA	Perfluorooctanesulfonamide	498 > 78	0.02	40	32	9
13C8-FOSA	IDA	506 > 78	0.02	48	32	9
PFDoA	Perfluorododecanoic acid	613 > 569	0.02	15	14	11
13C2-PFDoA	IDA	615 > 570	0.02	16	12	11
PFTrDA	Perfluorotridecanoic acid	663 > 619	0.02	12	12	11
PFTeDA	Perfluorotetradecanoic acid	713 > 669	0.02	12	18	11
PFHxDA	Perfluorohexadecanoic acid	813 > 769	0.02	18	15	12
PFODA	Perfluorooctadecanoic acid	913 > 869	0.02	20	16	12
13C2-PFTeDA	IDA	715 > 670	0.02	15	15	11
13C2-PFHxDA	IDA	815 > 770	0.02	18	15	12
EtFOSA	N-ethylperfuoro-1-octanesulfonamide	526 > 169	0.02	45	36	11
d5EtFOSA	IDA for EtFOSA	531 >169	0.02	40	30	11
MeFOSA	N-methylperfuoro-1-octanesulfonamide	512 > 169	0.02	45	25	11
d5MeFOSA	IDA	515 > 169	0.02	40	30	11
EtFOSAA	N-ethylperfluoro-1-octanesulfonamidoacetic acid	584 > 419	0.02	35	20	9
d5-EtFOSAA	IDA	589 > 419	0.02	30	25	9
MeFOSAA	N-methylperfluoro-1-octanesulfonamidoacetic acid	570 > 419	0.02	30	28	9
d5-MeFOSAA	IDA	573 > 419	0.02	30	25	9
6:2FTS	1H,1H,2H,2H-perfluorooctane sulfonate	427 > 407	0.02	40	30	5
M2-6:2FTS	IDA	429 > 409	0.02	40	28	5
8:2FTS	1H,1H,2H,2H-perfluorodecane sulfonate	527 > 507	0.02	40	28	8
M2-8:2FTS	IDA	529 > 509	0.02	40	28	8

Native Compounds	Native RT (minutes)	IS analog	IS RT (minutes)	Quantitation Method
PFBA	4.77	13C4-PFBA	4.79	Isotope Dilution
PFPeA	5.90	13C5-PFPeA	5.92	Isotope Dilution
PFBS	6.01	18O2-PFHxS	8.64	IS calculation
PFHxA	7.22	13C2-PFHxA	7.25	Isotope Dilution
PFHpA	8.57	13C4-PFHpA	8.59	Isotope Dilution
PFHxS	8.60	18O2-PFHxS	8.64	Isotope Dilution
PFOA	9.80	13C4-PFOA	9.83	Isotope Dilution
PFHpS	9.80	13C4-PFOS	10.90	IS calculation
PFNA	10.88	13C5-PFNA	10.92	Isotope Dilution
PFOS	10.87	13C4-PFOS	10.90	Isotope Dilution
PFDA	11.82	13C2-PFDA	11.86	Isotope Dilution
FOSA	12.41	13C8-FOSA	12.46	Isotope Dilution
PFDS	12.57	13C4-PFOS	10.90	IS calculation
PFUdA	12.62	13C2-PFUdA	12.66	Isotope Dilution
PFDoA	13.32	13C2-PFDoA	13.34	Isotope Dilution
PFTrDA	13.91	13C2-PFDoA	13.34	IS calculation
PFTeDA	14.39	13C2-PFDoA	13.34	IS calculation
PFHxDA	15.16	13C2-PFDoA	13.34	IS calculation
PFODA	15.57	13C2-PFDoA	13.34	IS calculation
EtFOSA	14.13	d-EtFOSA	14.11	Isotope Dilution
MeFOSA	13.73	d-MeFOSA	13.73	Isotope Dilution
EtFOSAA	12.63	D5-EtFOSAA	12.62	Isotope Dilution
MeFOSAA	12.3	D3-MeFOSAA	12.28	Isotope Dilution
6:2FTS	10.08	M2-6:FTS	10.08	Isotope Dilution
8:2FTS	11.95	M2-8:FTS	11.95	Isotope Dilution

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Shimadzu HPLC

Routine Instrument Operating Conditions					
HPLC Conditions (Shimadzu HPLC)					
Column (Column temp = 45°C)	Phenomenex Gemini 3 µm C18 110Å, 50 X 2 mm				
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol				
Gradient Program	**Time**	**%A**	**%B**		**Flow Rate mL/min.**
	0	90	10		0.60
	0.1	45	55		0.60
	4.5	1	99		0.60
	4.95	1	99		0.60
	5	90	10		0.60
	Maximum Pressure limit = 5,000 psi				
Injection Size	2 µL (fixed amount throughout the sequence)				
Run Time	~6.6 minutes				
Mass Spectrometer Interface Settings (SCIEX 5500)					
MS Interface Mode	ESI Negative Ion				
Ion Spray Voltage (kV)	4.5				
Entrance Potential (V)	5				
Declustering Potential (V)	25				
Desolvation Temp	600ºC				
Curtain Gas	35 psi				
Collision Gas	8 psi				

Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	RT (Min)
PFBA	Perfluorobutanoic acid	212.9 > 169	0.011	-5	-12	-25	-31	1.74
13C4_PFBA	IDA	217 > 172	0.011	-5	-12	-25	-31	1.74
PFBS	Perfluorobutanesulfonic acid	298.9 > 80	0.011	-6	-58	-55	-37	1.76
PFBS_2	Perfluorobutanesulfonic acid	298.9 > 99	0.011	-5	-40	-55	-12	1.76
PFPeA	Perfluoropentanoic acid	262.9 > 219	0.011	-7	-12	-20	-34	1.99
13C5_PFPeA	IDA	267.9 > 223	0.011	-7	-12	-20	-35	1.99
PFHxA	Perfluorohexanoic acid	313 > 269	0.011	-5	-12	-25	-37	2.25
13C2_PFHxA	IDA	315 > 270	0.011	-5	-12	-25	-38	2.25
PFHpA	Perfluoroheptanoic acid	363 > 319	0.011	-6	-12	-25	-41	2.57
13C4_PFHpA	IDA	367 > 322	0.011	-6	-12	-25	-41	2.57

PFHxS	Perfluorohexanesulfonic acid	399 > 80	0.011	-12	-74	-60	-43	2.59
18O2_PFHxS	IDA	403 > 84	0.011	-12	-74	-60	-43	2.59
6:2 FTS	Sodium 1H,1H,2H,2H-perfluorooctane sulfonate	427 > 407	0.011	-7	-32	-50	-10	2.91
M2-6:2FTS	IDA	429 > 409	0.011	-7	-32	-50	-10	2.91
PFOA	Perfluorooctanoic acid	413 > 369	0.011	-6	-14	-25	-44	2.93
PFOA_2	Perfluorooctanoic acid	413 > 169	0.011	-5	-22	-25	-12	2.93
13C4_PFOA	IDA	417 > 372	0.011	-6	-14	-25	-44	2.93
PFHpS	Perfluoroheptanesulfonic acid	449 > 80	0.011	-11	-88	-65	-46	2.94
PFNA	Perfluorononanoic acid	463 > 419	0.011	-6	-14	-25	-47	3.29
13C5_PFNA	IDA	468 > 423	0.011	-6	-14	-25	-48	3.29
PFOS	Perfluorooctanesulfonic acid	499 > 80	0.011	-9	-108	-65	-50	3.29
PFOS_2	Perfluorooctanesulfonic acid	499 > 99	0.011	-5	-58	-65	-12	3.29
13C4_PFOS	IDA	503 > 80	0.011	-9	-108	-65	-50	3.29
PFDA	Perfluorodecanoic acid	513 > 469	0.011	-6	-16	-25	-51	3.65
13C2_PFDA	IDA	515 > 470	0.011	-6	-16	-25	-51	3.65
8:2 FTS	Sodium 1H,1H,2H,2H-perfluorodecane sulfonate	527 > 507	0.011	-7	-40	-50	-15	3.65
M2-8:2FTS	IDA	529 > 509	0.011	-7	-40	-50	-15	3.65
PFOSA	Perfluorooctanesulfonamide	498 > 78	0.011	-8	-85	-60	-50	3.7
13C8_PFOSA	IDA	506 > 78	0.011	-8	-85	-60	-50	3.7
N-MeFOSAA	N-methylperfluoro-1-octanesulfonamidoacetic acid	570 > 419	0.011	-7	-36	-40	-15	3.82
d3-MeFOSAA	IDA	573 > 419	0.011	-7	-36	-40	-15	3.82
PFDS	Perfluorodecanesulfonic acid	599 > 80	0.011	-11	-118	-85	-54	3.96
PFUdA	Perfluoroundecanoic acid	563 > 519	0.011	-7	-18	-25	-54	3.97
13C2_PFUdA	IDA	565 > 520	0.011	-7	-18	-25	-54	3.97
N-EtFOSAA	N-ethylperfluoro-1-octanesulfonamidoacetic acid	584 > 419	0.011	-7	-36	-50	-15	3.99
d5-EtFOSAA	IDA	589 > 419	0.011	-7	-36	-50	-15	3.99
MeFOSA	N-methylperfuoro-1-octanesulfonamide	512 > 169	0.011	-7	-37	-75	-15	4.21
d3MeFOSA	IDA	515 > 169	0.011	-7	-37	-75	-15	4.21
PFDoA	Perfluorododecanoic acid	613 > 569	0.011	-5	-18	-25	-54	4.3
13C2_PFDoA	IDA	615 > 570	0.011	-5	-18	-25	-54	4.3

EtFOSA	N-ethylperfuoro-1-octanesulfonamide	526 > 169	0.011	-7	-37	-75	-15	4.39
d5EtFOSA	IDA	531 > 169	0.011	-7	-37	-75	-15	4.39
PFTrDA	Perfluorotridecanoic acid	663 > 619	0.011	-7	-20	-25	-54	4.56
PFTeDA	Perfluorotetradecanoic acid	713 > 669	0.011	-2	-22	-25	-10	4.79
PFTeDA_2	Perfluorotetradecanoic acid	713 > 169	0.011	-7	-36	-25	-30	4.79
13C2_PFTeDA	IDA	715 > 670	0.011	-2	-22	-25	-10	4.79
PFHxDA	Perfluorohexadecanoic acid	813 > 769	0.011	-7	-24	-25	-54	5.25
13C2_PFHxDA	IDA	815 > 770	0.011	-7	-24	-25	-54	5.25
PFODA	Perfluorooctadecanoic acid	913 > 869	0.011	-7	-26	-25	-54	5.55

11.14.2. Tune and calibrate the instrument as described in Section 10.

11.14.3. A typical run sequence is as follows:

- Primer (A number of primers are injected for conditioning of the instrument before analysis, especially when the instrument was idled or changed from a different analysis).
- Blank
- Calibration Curve
- ICB
- ICV
- MB
- LCS
- LCSD (if applicable)
- Sample 1
- Sample 1 MS (if applicable)
- Sample 1 MSD (if applicable)
- Sample 2 (up to sample 10 before next CCV)
- CCV
- Up to 10 samples.
- End sequence with CCV

12. CALCULATIONS

12.1. If the concentration of the analyte ions exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. It may be necessary to dilute samples due to matrix.

12.2. Qualitative Identification

12.2.1. The retention times of PFC's with labeled standards must be the same as that of the labeled IDA's to within 0.05 min. For PFC's with no labeled standards, the RT must be within ± 0.3 minutes of the ICV and CCV standards. *Note: The IS RT and native RT may be offset by 0.02 to 0.04 minutes.*

12.3. The ICAL established in Section 10 is used to calculate concentrations for the extracts.

12.4. Extract concentrations are calculated as below. The first equation applies to the linear fit, the second to the quadratic line fit.

Equation 4

$$\text{Concentration, ng/mL} = \frac{y - c}{b}$$

Equation 5

$$\text{Concentration, ng/mL} = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where:

y = $\frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$
x = concentration
a = curvature
b = slope
c = intercept

12.5. Water Sample Result Calculation:

Equation 6

$$\text{Concentration, ng/L} = \frac{C_{ex} V_t}{V_o}$$

Where:

C_{ex} = Concentration measured in sample extract (ng/mL)
V_t = Volume of total extract (mL)
V_o = Volume of water extracted (L)

12.6. Soil Sample Result Calculation:

Equation 7

$$\text{Concentration, } ng/g = \frac{C_{ex} V_t}{W_s D}$$

Where ng/g = µg/kg and:

C_{ex} = Concentration measured in sample extract (ng/mL)

V_t = Volume of total extract (mL)
W_s = Weight of sample extracted (g)
D = Fraction of dry solids, which is calculated as follows:

$$\frac{100 - \%\ \text{moisture in sample}}{100}$$ (for dry weight result)

12.7. IDA Recovery Calculation:

Equation 8

$$\%\ \text{Recovery} = \frac{RF_{ex} A_t}{Amt}$$

Where ng/g = µg/kg and:

RF_{ex} = Response Factor for IDA compound
A_t = Area response for IDA compound
Amt = Amount spike of IDA

12.8. If results are to be reported as ammonium perfluorooctanoate (APFO), instead of PFOA, apply a multiplier of 1.0406 to the sample results to correct for the molecular weight differences between PFOA and APFO or this adjustment can be made during the preparation of the standards used for calibration. (Use one, not both.)

12.9. Raw data, calibration summaries, QC data, and sample results are reviewed by the analyst. These must also be reviewed thoroughly by a second qualified person. See the Data Review Policy (WS-PQA-0012). These reviews are documented on the Data Review Checklist.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration of Capability (IDOC)

Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

13.4. The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-0006 and policy WS-PQA-003.

14. POLLUTION PREVENTION

14.1. All waste will be disposed of in accordance with Federal, State and Local regulations.

14.2. Solid phase extraction used for water samples greatly reduces the amount of solvent used compared to liquid-liquid extraction.

14.3. Standards and reagents are purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.4. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.5. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless waste is being transferred.

14.6. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out:

15.1. Assorted test tubes, autovials, syringes, filter discs and cartridges. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the hazardous waste – landfill steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Extracted soil samples, used sodium sulfate, paper funnel filters, glass wool, thimbles, and extracted solids contaminated with solvents. Dump these materials into an orange contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the incineration steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Waste Methanol. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end

of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to no less than six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.4. Mixed water/methanol waste from soil extraction. Collect the waste in the HPLC waste carboy. When full, or after no more than one year, dump into the blue plastic HPLC collection drum in the H3 closet. When the drum is full, to no less than six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.

15.5. Aqueous acidic waste from the LCMS instrument contaminated with methanol. This is collected in a 1-gallon carboy at the instrument. When the carboy is full, or after no more than one year, it is emptied into the blue plastic HPLC collection drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.

15.6. Autovials contaminated with methanol. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After all autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methanol and other contaminants to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES

16.1. Cheryl Moody, Wai Chi Kwan, Johnathan W. Martin, Derek C. G. Muir, Scott A. Mabury, “Determination of Perfluorinated Surfactants in Surface Water Samples by Two Independent Analytical Techniques: Liquid Chromatography/Tandem Mass Spectrometry and 19FNMR,” Analytical Chemistry 2001, 73, 2200-2206.

16.2. John Giesy et al., “Accumulation of Perfluorooctane Sulfonate in Marine Mammals”, Environmental Science & Technology, 2001 Vol. 35, No. 8, pages 1593-1598.

16.3. U.S. EPA, “Residue Chemistry Test Guidelines, OPPTS 860.1340, Residue Analytical Method”, EPA 712-C-95-174, August 1995.

16.4. STL Denver White Paper DEN-W-LC-002, “Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)”, Mark Dymerski, September 5, 2003.

16.5. STL Denver White Paper DEN-W-LC-003, “Addendum A to Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance

Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, August 6, 2003.

16.6. STL Denver White Paper DEN-W-LC-004, "Method Validation Study for Analysis of Perfluorooctanoic Acid in Waters by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, January 26, 2005.

16.7. Waters application note; "Acquity UPLC System for Quantifying Trace Levels of Perfluorinated Compounds with an Acquity PFC Analysis Kit", Peter J. Lee, Evan T. Bernier, Gordon T. Fujimoto, Jeremy Shia, Michael S. Young, and Alice J. Di Gloia, Waters Corporation, Milford, MA. USA.

16.8. US EPA, "Method 537 - Determination of Selected Perfluorinated alkyl acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometery (LC/MS/MS)", Version 1.1, September 2009, J.A. Shoemaker, P.E. Grimmett, B.K. Boutin, EPA Document #: EPA/600/R-08/092

17. METHOD MODIFICATIONS

Modifications from Method 537 are detailed below:

17.1. Water sample containers are not preserved with Trizma.

17.2. The method has been modified to address soil/solid matrices. The extraction holding time is set at 14 days.

17.3. The analyte list has been expanded. The number of labeled analytes has been expanded as well to improve quantitation.

17.4. The reporting limits differ as they are all set at one consistent value.

17.5. Calibration levels differ from the referenced method.

17.6. More labeled analytes are fortified into the samples prior to the extraction process. Most target analytes are quantitated against a labeled analyte.

17.7. There is no symmetry requirement.

17.8. Calibration, both initial and continuing, has different acceptance criteria due to the longer list of analytes, and the use of IDA/external standard quantitation.

17.9. The eluents and HPLC configuration differs. As a result the final extract is in 80:20 methanol: water.

17.10. The LCS and MS/MSD are spiked at one concentration and do not rotate between a low to high levels.

17.11. Samples are not checked for residual chlorine or pH.

17.12. A different SPE cartridge (Waters OASIS WAX) is used for the extraction process. As a result solvents and elution procedures are different.

18. ATTACHMENTS

18.1. Attachment 1 - Analysis of Perfluorinated Compounds (PFCs) in Water via Direct Aqueous Injection (DAI).

19. REVISION HISTORY

19.1. WS-LC-0025, Revision 2.1, Effective 12/09/2016

19.1.1. Section 8.2, second sentence, changed 7 days to 14 days.

19.1.2. Note following Section 8.2, changed to read: "NOTE: As of this writing, Method 537 provides for a 14 day holding time for water samples preserved with Trizma buffer. The scientific literature indicates that perfluorinated substances are highly persistent in the environment. TestAmerica Sacramento has conducted holding time studies that support a 14 day holding time for aqueous samples with and without Trizma preservation. TestAmerica Denver has conducted stability studies indicating that medium- and low-level solutions of PFOA are stable for at least three months in polystyrene and polypropylene plastics at 0-6C. The 14 day/40 day holding times given above are based on the holding time study and general EPA convention for the holding time of extractable organic compounds in water and soil."

19.1.3. Section 17.1, removed the second sentence "Holding time has been changed to 7 days for extraction."

19.1.4. Editorial Changes

19.2. WS-LC-0025, Revision 2.0, Effective 11/18/2016

19.2.1. Replace "internal standard" with "IDA" throughout SOP.

19.2.2. Section 4.7, changed last sentence of paragraph to include "…in the standard **and/or sample** must…".

19.2.3. Section 6.9.4, added - "Phenomenex Gemini 3 µm C18 110Å, 50 X 2 mm, Part No. 00B-4439-B0."

19.2.4. Section 6.9.5, added – "Phenomonex Luna 5 µm C18(2) 100Å, 30 X 3 mm, Part No. 00A-4252-Y0."

19.2.5. Section 6.14, added – "SCIEX 5500 Triple Quad MS. The system utilizes Chrom Peak Review, version 2.1 or equivalent."

19.2.6. Section 7.4.1, added - "A technical (qualitative) grade PFOA standard is analyzed after an initial calibration initially, when a new column is installed or when significant changes are made to the HPLC parameters. This solution is used as a reference for the PFOA isomers (branched and linear) retention times."

19.2.7. Section 11.14.1, HPLC settings, gradient time 1, corrected flow rate from 0330 mL/min to 0.30 mL/min.

19.2.8. Section 11.14.1, MS Settings, removed the following lines for Waters instrument and added additional Table for Shimadzu HPLC.

PFPeS	Perfluoropentanesulfonate	3749 > 80	0.02	55	32	3
PFNS	Perfluorononanesulfonate	549 > 80	0.02	65	54	8
PFDoS	Perfluorododecanesulfonate	699 > 80	0.02	80	55	11

19.2.9. Section 15, added – "Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent."

19.2.10. Editorial chamges.

19.3. WS-LC-0025, Revision 1.9, Effective 05/27/2016

19.3.1. Editorial Changes.

19.3.2. Added Attachment 1.

19.4. WS-LC-0025, Rev 1.8, Effective 05/19/2016

19.4.1. Section 1.2, changed water sample volume from 500 to 250 mL.

19.4.2. Section 7.6, change the LCS solution from 1000 ng/mL to 500 ng/mL

19.4.3. Section 8.1, changed the sample container from 500 mL volume to 250 mL volume for water samples.

19.4.4. Section 11.3.2, 11.3.4, and 11.4.5, changed water sample volume from 500 to 250 mL.

19.4.5. Section 11.3.5, change the volume of spike added for EtFOSA/MeFOSA from 100 uL to 200 uL.

19.4.6. Section 11.3.6, change the volume of solution added from 0.050mL (50 uL) to 0.025 ml (25 ul), and from 250 to 125 uL if EtFOSA/MeFOSA is requested.

19.4.7. Section 11.7.2, change the volume of methanol from 800 uL to 400 uL.

19.4.8. Section 11.7.3, change the volume of water added from 200 uL to 100 uL.

19.4.9. Section 11.8.4, changed the volume of spike added from 20 uL to 40 uL.

19.4.10. Editorial Changes

19.5. WS-LC-0025 Rev. 1.7, Effective 03/18/2016

19.5.1. Section 4.5 – Deleted the last sentence in this section: "Until more information is available" and changed "excluded" to "included".

19.5.2. Section 4.7 - Deleted the last sentence. "Chromatographic peaks in a sample must be integrated in the same way as the CAL standard."

19.5.3. Section 7.4 – Changed upper calibration limit (CS-7) for all analytes from 500 ng/mL to 400 ng/mL

19.5.4. Section 9.8.2 – Revised 1st sentence to "IDA recoveries are flagged if they are outside of the acceptance limits (25–150%) "

19.5.5. Section 11.3.5 – Added to end of Section, "If EtFOSA and/or MeFOSA are required, increase the amount of LCS Matrix PFC spike solution added to 100 uL."

19.5.6. Editorial changes.

19.6. WS-LC-0025 Rev. 1.6, Effective January 22, 2016

19.6.1. Section 11.6.1 – Revised to include rinse of sample container

19.6.2. Section 11.6.2 – Revised to include rinse of sample container.

19.6.3. Editorial changes

19.7. WS-LC-0025 Rev. 1.5, Effective November 1, 2015

19.7.1. Added the analytes EtFOSA, MeFOSA, EtFOSAA, MeFOSAA, 6:2FTS and 8:2FTS to Sections 1.1, 7.4 and 11.14.

19.7.2. Added Sections 2.5, 10.9.4 and 12.7 to address external standard quantitation.

19.7.3. Section 9.8 was updated and Section 12.7 added to address the calibration and quantitation of IDA compounds.

19.7.4. Added verbiage to Section 11 to address specifics if EtFOSA and/or MeFOSA are requested.

19.7.5. Added Section 11.3.7 for the specific processing of aqueous samples for EtFOSA and/or MeFOSA.

19.7.6. Added analytes and pertinent information to Section 11.14.1.

19.7.7. Editorial changes

19.8. WS-LC-0025 Rev. 1.4, Effective August 28, 2015

19.8.1. Updated copyright statement on cover page.

19.8.2. Section 1.1 – Renamed sulfonates to sulfonic acids and corrected the CAS# for PFHpS and PFOS. Removed FOSSA from table.

19.8.3. Section 7.4 – Renamed sulfonates to sulfonic acids and removed FOSSA remark.

19.8.4. Section 6.18 – Routine and Preventative maintenance table added.

19.8.5. Added Section(s) 17.1 thru 17.13 - Method modifications to Method 537.

19.8.6. Editorial changes.

19.9. WS-LC-0025 Rev. 1.3, Effective Oct. 31, 2014

19.9.1. Removed references to glass containers in Section 8.1, 8.2 and Notes following Section 8.

19.9.2. Editorial changes

19.10. WS-LC-0025 Rev. 1.2, Effective July 5, 2013

19.10.1. Updated Tables in Section 11.14 with current specifications.

19.10.2. Editorial changes.

19.11. WS-LC-0025 Rev. 1.1, Effective May 25, 2012

19.11.1. Editorial revisions.

19.12. WS-LC-0025 revision 1.0, Effective May 3, 2011

19.12.1. This is the original SOP. SOP was created from WS-LC-0020.

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water samples via direct aqueous injection (DAI) for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a SCIEX 5500.

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1

1.2. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	1.0 mL	2.0 ng/L	2 to 200 ng/L

2. SUMMARY OF METHOD

2.1. A 1 mL aliquot of sample is diluted to a 40:60 methanol:water extract and analyzed by LC/MS/MS. PFCs are separated from other components on a C18 column with a solvent gradient program using 20mM Ammonium Acetate/water and methanol.

3. DEFINITIONS

Refer to Section 3 of the main body of this SOP for a summary of definitions.

4. INTERFERENCES

Refer to Section 4 of the main body of this SOP for interferences.

5. SAFETY

Refer to Section 5 of the main body of this SOP for safety information.

6. EQUIPMENT AND SUPPLIES

Refer to Section 6 of the main body of this SOP for supplies, other than those listed below specific to the DAI analysis.

6.1. 2 mL auto sampler vials, clear glass, Thermo Scientific Nation surestop vial, part no. C5000-1, or equivalent**.**

6.2. Vial caps, Thermo Scientific National AVCS blue cap, pre slit TEF/STL septa, part no. C5000-55B or equivalent.

6.3. Eppendorf 1000 uL epTIPS, part no. 022491954 or equivalent.

6.4. Eppendorf 200 uL epTIPS, part no. 022491938 or equivalent.

6.5. 50 mL graduated plastic centrifuge tubes, SCP Science DigiTUBES part no. 010-500-263 or equivalent

The 5 items above are to be maintained in the drawer labeled "Segregated Supplies for DAI Analysis" in the LC/MS instrument room

6.6. 1000 uL Pipette: Eppendorf Research Plus

6.7. 100 uL Pipette: Rainin EDP3-Plus

6.8. 250 mL HDPE bottles with PPE screw caps, ESS part no. 0250-1902-QC or equivalent.

6.9. Analytical columns

6.9.1. Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm, Part No. 00D-4439-Y0, or equivalent.

6.9.2. PFC Isolator column, Phenomenex Luna C18 5 um, 50 mm x 4.6 mm, part no. 00B-4252-E 0 or equivalent.

6.10. SCIEX 5500 Triple Quad MS. The system utilizes Chrom Peak Review, version 2.1 or equivalent.

6.11. Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.

7. REAGENTS AND STANDARDS

Refer to Section 7 of the main body of this SOP for reagents and standards, other than those listed below specific to the DAI analysis.

7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1. Ammonium acetate, Fisher Optima LCMS grade (20 mM in water), part no. A114-50, or equivalent.

7.1.2. Methanol, Baker HPLC grade, part no. 9093-03.

7.1.3. Water, Nanopure or Millipore or Fisher Optima LCMS grade, part no. W6-4, must be free of interference and target analytes.

7.2. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of the stock solutions (Section 7.2 of the main body of this SOP) in 40:60 methanol:water. The calibration stock solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.3. Initial Calibration (ICAL) Levels (ng/L)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7	CS-8
Perfluoroalkylcarboxylic acids (PFCAs)								
PFHpA	1.0	2.0	5.0	10	20	50	100	200
PFOA	1.0	2.0	5.0	10	20	50	100	200
PFNA	1.0	2.0	5.0	10	20	50	100	200
Perfluorinated sulfonic acids (PFSAs)								
PFBS	1.0	2.0	5.0	10	20	50	100	200
PFHxS	1.0	2.0	5.0	10	20	50	100	200
PFOS	1.0	2.0	5.0	10	20	50	100	200
Labeled Isotope Dilution Analytes (IDA)								
13C4-PFHpA	50	50	50	50	50	50	50	50
13C4-PFOA	50	50	50	50	50	50	50	50
13C5-PFNA	50	50	50	50	50	50	50	50
18O2-PFHxS	50	50	50	50	50	50	50	50
13C4-PFOS	50	50	50	50	50	50	50	50

Note- The above calibration levels are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.

7.4. LCS/Matrix PFC Spike Solution, 100 ng/mL.

The PFC spike solution is prepared by diluting all PFCs to produce a solution containing PFCs each at 100 ng/mL in methanol.

7.5. PFC Isotope Dilution Analyte (IDA) Spike Solution, 1 ng/mL.

The PFC-IDA solution is prepared by diluting all labeled PFCs to produce a solution each at 1 ng/mL in methanol.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6 °C for shipment to the laboratory.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6 °C Water samples must be analyzed within 28 days of collection.

9. QUALITY CONTROL

Refer to Section 9 of the main body of this SOP for Quality Control information.

10. CALIBRATION

Refer to Section 10 of the main body of the SOP for calibration information.

11. PROCEDURE

Refer to Section 11 of the main body of this SOP for procedures, other than those listed below specific to the DAI analysis.

11.1. Water Sample Preparation

11.1.1. Visually inspect samples for the presence of settled and or suspended sediment. If sediment is apparent, remove the aliquot for testing from the aqueous layer. If the sediment concentration is too high centrifuge the sample first or filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration.

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.1.2. Prepare an LCS and method blank by adding 250 mL of HPLC grade water into a 250 mL HDPE bottle.

11.1.3. If requested, find the client assigned sample for MS/MSD.

11.1.4. Spike directly into the sample bottles for the LCS and MS/MSD (if requested) with 0.050 mL (50 uL) of the LCS/Matrix PFC Spike solution (Section 7.4). This will result in a sample concentration of 20 ng/L. Shake well to disperse spike.

11.1.5. Measure 1 mL of each sample using an Eppendorf pipette and pour into a

labeled 2.0 mL injection vial. This includes the LCS and method blank samples as well.

11.1.6. Be sure to "prepare" the pipette by collecting two 1 mL aliquots and disposing of them, and then collect the aliquot for testing.

11.1.7. Add 83 uL of surrogate solution (PFC IDA Spike Solution, Section 7.5) into each vial for each sample and QC sample. This will result in an extract concentration of 50 ng/L for the surrogate.

11.1.8. Add 577 uL of methanol to each sample for a final solvent composition of 40:60 methanol:water.

11.1.9. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFCs.

11.1.10. Vortex to mix the mixture well.

11.2. Instrument Analysis

11.2.1. Suggested operation conditions are listed below:

Routine Instrument Operating Conditions					
HPLC Conditions (Shimadzu HPLC)					
Column (Column temp = 35°C)	Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm				
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol				
Gradient Program	**Time (min)**	**%A**	**%B**	**Curve**	**Flow Rate (mL/min)**
	0	90	10	6	0.60
	1	90	10	6	0.60
	1.5	35	65	6	0.60
	8	5	95	6	0.60
	8.1	1	99	6	0.60
	12	1	99	6	0.60
	12.5	90	10	6	0.60
	Maximum Pressure limit = 5,000 psi				
Injection Size	950 uL (fixed amount throughout the sequence)				
Run Time	17.1 minutes				

Mass Spectrometer Interface Settings (SCIEX 5500))	
MS Interface Mode	ESI Negative Ion
Ion Spray Voltage (kV)	4.5
Entrance Potential (V)	5
Declustering Potential (V)	25
Desolvation Temp	550 ºC
Curtain Gas (nitrogen) Flow	35 psi
Collision Gas (nitrogen) Flow	8 psi

Mass Spectrometer Scan Settings						
Compound	**Comments**	**Reaction (MRM)**	**Dwell (sec)**	**Ent. Pot. (V)**	**Col. Energy (V)**	**Declu. Pot. (V)**
PFBS	Perfluorobutanesulfonate	299 > 80	0.02	6	58	55
18O2-PFHxS	IS	403 > 84	0.02	12	74	60
PFHpA	Perfluoroheptanoic acid	363 > 319	0.02	6	12	25
13C4-PFHpA	IS	367 > 322	0.02	6	12	25
PFHxS	Perfluorohexanesulfonate	399 > 80	0.02	12	74	60
18O2-PFHxS	IS	403 > 84	0.02	12	74	60
PFOA	Perfluorooctanoic acid	413 > 369	0.02	6	14	25
13C4PFOA	IS	417 > 372	0.02	6	14	25
PFNA	Perfluorononanoic acid	463 > 419	0.02	6	14	25
13C5-PFNA	IS	468 > 423	0.02	6	14	25
PFOS	Perfluorooctanesulfonate	499 > 80	0.02	9	108	65
13C4-PFOS	IS	503 > 80	0.02	9	108	65

Native Compounds	**Native RT (minutes)**	**IS analog**	**IS RT (minutes)**	**Quantitation Method**
PFBS	6.68	18O2-PFHxS	7.76	IS calculation
PFHpA	7.77	13C4-PFHpA	7.77	Isotope Dilution
PFHxS	7.76	18O2-PFHxS	7.76	Isotope Dilution
PFOA	8.44	13C4-PFOA	8.44	Isotope Dilution
PFNA	9.10	13C5-PFNA	9.10	Isotope Dilution
PFOS	9.06	13C4-PFOS	9.06	Isotope Dilution

11.2.2. Tune and calibrate the instrument as described in Section 10.

12. CALCULATIONS

Refer to Section 12 of the main body of this SOP for calculation information.

13. METHOD PERFORMANCE

Refer to Section 13 of the main body of this SOP for method performance information.

14. POLLUTION PREVENTION

Refer to Section 14 of the main body of this SOP for pollution prevention information.

15. WASTE MANAGEMENT

Refer to Section 15 of the main body of this SOP for waste management information.

16. REFERENCES

Refer to Section 16 of the main body of this SOP for reference information.

17. METHOD MODIFICATIONS

Refer to Section 17 of the main body of this SOP for modifications from Method 537, except as detailed below:

17.1. Water samples are prepared at 1.0mL, not 250mL.

17.2. Water sample containers are not preserved with Trizma. Holding time has been changed to 28 days for analysis.

17.3. The eluents and HPLC configuration differs. As a result the final extract is in 40:60 methanol: water.

18. ATTACHMENTS

There are no attachments to this SOP.

19. REVISION HISTORY

19.1. WS-LC-0025, Revision 2.1, Effective 12/09/2016

19.1.1. Revised section 8.2. Changed the holding time for water samples from 7 to 14 days.

19.1.2. Revised section 17.1. Removed the line “Holding time has been changed to 7 days for extraction.”

19.2. WS-LC-0025 Attachment 1, Revision 1.9, Effective 05/27/2016

19.2.1. This is the first version of this Appendix.

Appendix D

Contractor PFAS SOPs

LIST OF FIELD STANDARD OPERATING PROCEDURES (SOPS)

SOP #1 – SITE RECONNAISSANCE, PREPARATION, AND RESTORATION PROCEDURES

1.1 SITE ACCESS

Parsons will obtain any required security badges for Parsons and subcontractor personnel working on this project, as well as any vehicle passes that are required. In addition, Parsons will also provide SEDA with a minimum of one week notice prior to requiring access to any secure sites. However, there may be instances where one week notice is not feasible given project-specific requirements; these will be addressed on a case-by-case basis and as much notice as possible will be provided to SEDA.

1.2 UTILITY LOCATION

Areas designated for intrusive activities will be assessed for the presence of underground utilities. In addition, Dig Safely New York will be contacted at least 48 hours prior to intrusive activities to obtain a routine ticket for utility location. Dig Safely New York ticket requests will be made by calling 811 (if inside New York) or 800-962-7962 if outside of New York or by placing a request online at http://www.digsafelynewyork.com using i-Notice. Based on the type of investigation, additional methods to identify utilities may be used including geophysical survey, hand probes, and line tracing.

1.3 ESTABLISHMENT OF DECONTAMINATION AREA

A centralized decontamination area will be established in an area designated by SEDA for drilling rigs and equipment if necessary. The decontamination area will be large enough to allow storage of cleaned equipment and materials prior to use, as well as to stage drums of decontamination waste if generated. The decontamination area will be lined with heavy-gauge plastic sheeting, and designed with a collection system to capture decontamination waters and steam condensate. Solid wastes will be accumulated in United States (U.S) Department of Transportation (DOT) approved 55-gallon drums and subsequently transported to a waste storage area designated by SEDA. Smaller decontamination areas for portable equipment will be provided as necessary. These locations will include basins, tubs, or buckets to capture decontamination fluids.

1.4 SITE RESTORATION

Each work site or sampling location will be restored to its original condition whenever possible. Efforts will be made to minimize impacts to work sites and sampling locations, particularly those in or near sensitive environments such as wetlands. Following the completion of work at a site; drums, trash, and other waste generated from the work process will be removed. Decontamination and/or purge water and soil cuttings will be transported to designated locations. Site restoration will also consist of repair of tire ruts and installation of topsoil and an appropriate seed mix when necessary.

SOP #2 – EQUIPMENT DECONTAMINATION

Equipment that may directly or indirectly contact samples will be decontaminated in a designated decontamination area. This includes auger flights, direct push rods, sampling devices, and instruments, such as sounders. In addition, care will be taken to prevent the sample from coming into contact with potentially contaminating substances, such as tape, oil, engine exhaust, corroded surfaces, and dirt.

2.1 DIRECT PUSH AND SOIL SAMPLING DEVICES

The following procedure will be used to decontaminate soil sampling and drilling devices, such as direct-push rods that can be hand-manipulated. For sampling and smaller drilling devices equipment decontamination will be performed by scrubbing the equipment with a solution of potable water and Alconox or equivalent laboratory-grade detergent. Then rinse the equipment with copious quantities of potable water followed by distilled water. As appropriate, decon may be conducted using PFAS-free water provided by the laboratory in place of distilled water to minimize potential contamination of the sample. Air dry the equipment on a clean surface or rack elevated above the ground surface. If the sampling device will not be used immediately after being decontaminated, it will be wrapped in oil-free aluminum foil.

2.2 GROUNDWATER MONITORING EQUIPMENT

All groundwater sampling equipment will be dedicated or non-contact in nature. Therefore, no groundwater monitoring equipment decontamination procedures are necessary.

However, any decontamination that is conducted will follow SOP #2, Section 3.2.2.

SOP #3 – GROUNDWATER SAMPLE COLLECTION

3.1 SAMPLE COLLECTION PROCEDURES

The following procedure will be followed for the collection of groundwater samples:

1. Record comments pertinent to the color and any obvious odors associated with the water;
2. Arrange and label necessary sample bottles and ensure that preservatives are added, as required. Labeling must include a unique sample number, date of sampling, the initials of the sampling personnel, and the identity of the sample fraction. Additionally, provide any information pertinent to the preservation materials or chemicals used in the samples;
3. Samples shall be collected using high density polyethylene (HDPE) tubing. Purging can be performed either by fitting the tubing with a check valve and manually pumping or by using a peristaltic pump.
4. Immediately seal each sample and place it on ice in a cooler to maintain sample integrity. Do not allow the samples to freeze, as the bottles may break;
5. Once sampling is completed, recover GeoProbe SP-22 groundwater sampler or temporary monitoring well and sample tubing and dispose;
6. Clean up and remove any debris left from the sampling event. Be sure that wastes are properly containerized and labeled;
7. Review sampling records. Ensure that necessary data is completed. Add additional information as may be needed.

3.2 INFORMATION AND SPECIAL PRECAUTIONS SPECIFIC TO PER/POLY FLUORINATED ALKYL SUBSTANCES (PFAS)

3.2.1 Prohibited and Acceptable Items

Required laboratory detection limits for the analysis of PFAS are extremely low and the use of PFAS in everyday products is widespread leading to many sources of potential trace contamination. Field personnel are expected to avoid the use of all products treated with PFAS while on site.

General precautions to follow products to avoid while on-site include the following. Note that this is not an exhaustive list. A summary of prohibited and acceptable items for sampling PFAS is provided in Table 1.

Food Related

- Paper food packaging is often treated with PFAS to resist wetting. As such, personnel should avoid paper bags, paper food packaging, paper wrapping (e.g. sandwich wrap), paper beverage cups, as well as other coated papers.
- Aluminum foil should not be used on site
- Food that has been fried in a frying pan due to the potential for contamination from Teflon-coated cooking surfaces.

- Coated textiles of any type should be used on site
- Snacks and meals should not be eaten in the field vehicle or in the work area. Field personnel should leave the work area by a minimum of 10 meters (downwind whenever possible) when taking breaks for food and beverages.

Field Gear/Clothing

- Water resistant, water proof, or stain treated clothing such as Gore-Tex should not be worn by field personnel. Clothing worn during field sampling should be made of natural fiber such as cotton or wool.
- Clothing made of synthetic fibers. Clothing worn during field sampling should be made of natural fiber such as cotton or wool.
- Field clothing should ideally be old and well laundered.
- Field clothing should be washed using a minimal use of unscented detergent and no fabric softener or other additives. Once clean the clothing should be washed again in water only before drying. No fabric conditioner or dryer sheets should be used while drying.
- Rite in the Rain field notebooks/paper and similar products are not be used. Field records should be recorded on loose uncoated paper.
- Field notes and records should be made in pencil. Ballpoint pens and markers are not to be used for notes. Rite for Rain Pens or pencil may be used for samples labels. Sample labels may also be pre-printed by the laboratory; if pencil is used to write on the sample labels, those bottles will be double bagged.
- Clipboards should be made of Masonite or aluminum. Plastic clipboards, binders, and spiral bound notebooks are not acceptable.
- Safety toe boots made from synthetic fibers and treated for water resistance are acceptable for use in order to maintain personnel protection. However, all contact with the boots is to be made at least 10 meters away from the work area. New gloves are to be donned prior to making contact with the boots and are to be disposed immediately afterwards. Boots containing Gore-Tex and/ or Tyvek are not to be used on site.
- Disposable nitrile gloves must be worn at all times. A new set of gloves will be donned prior to conducting any of the following activities at each sample location:
    - Equipment decontamination,
    - Contact with bottleware and/or PFAS free water containers,
    - Insertion of anything into the well (e.g. samplers, tubing, etc…),
    - Insertion of silicone tubing into peristaltic pump,
    - Completion of well purge, prior to sample collection,
    - Collection/handling of QC/QA samples,

  - Following handling any non-dedicated field equipment, contact with non-decontaminated surfaces, and
  - When deemed necessary by field personnel.
- Vehicle seating is often treated with stain resistant products. Therefore, direct contact with vehicle seats should be avoided by covering each seat with a well laundered cotton sheet for the duration of the sampling event.
- Samples should be kept iced using only regular water ice double-bagged in polyethylene bags. No chemical ice packs (blue ice) are allowed.

Personal Hygiene

- On the day of sampling, field personnel should not use shampoo, conditioner, body gel, cosmetics, or cosmetic/hand/body creams as part of their personal hygiene routine. The use of bar soap is acceptable; however, bar soaps containing additional moisturizers should be avoided,
- It is recommended that field personnel shower the night before the sampling event and rinse with water only the morning of the sampling event,
- Cosmetics, moisturizers, sunscreens, insect repellant, and dental floss, except for those in Table 1, shall not be used on or off site throughout the duration of the field sampling program,
- For restroom breaks, field personnel shall move at least 10 meters from the work area before removing gloves and overalls. Personnel should wash their hands as normal allowing for extra time for rinsing after soap use. It is preferred to dry hands after washing using a hand dryer rather than paper products whenever possible.

Site Visitors

- All visitors to the site are to be asked to remain a minimum distance of at least 10 meters from all sampling areas.

Rain Events

- The use of waterproof rain gear is not permitted while sampling. Therefore, field sampling will not take place in the presence of persistent rainfall. Field gear shall be removed from the sampling area during rainfall and can be returned after the rain subsides.
- The use of a waterproof gazebo tent is acceptable for use to provide shelter from the rain if the schedule does not allow for work to stop during rain. The gazebo should be erected directly overtop of the sampling area taking precautions that water running off of the gazebo does not enter into work areas. Since a waterproof gazebo represents a potential for PFAS cross-contamination precautions should be taken when using them. Gloves should be donned whenever contact with the gazebo is made and the gloves should be disposed of immediately following contact.

Table 1: Summary of Prohibited and Acceptable Items for Sampling of PFCs

PROHIBITED ITEMS	ACCEPTABLE ITEMS
Field Equipment	
Teflon® containing materials	High-density polyethylene (HDPE) materials
Low density polyethylene (LDPE)	Acetate liners
Aluminum foil	Silicon tubing
Waterproof field books	Loose paper (non-waterproof)
Plastic clipboards, binders, or spiral hard cover notebooks	Aluminum field clipboards or with Masonite
	Rite for Rain pens®
Post-It Notes	
RE-usable Chemical (blue) ice packs	Regular ice in polyethylene bags (double bagged)
Field Clothing and Personal Protective Equipment (PPE)	
New cotton clothing or synthetic water resistant, waterproof, or stain-treated clothing, clothing containing Gore-Tex™	Well-laundered clothing, defined as clothing that has been washed 6 or more times after purchase, made of natural fibers (preferable cotton)
Clothing laundered using fabric softener	No fabric softener
Boots containing Gore-Tex™ Tyvek®	Boots made with polyurethane and polyvinyl chloride (PVC) Cotton Clothing
No cosmetics, moisturizers, hand cream, or other related products as part of personal cleaning/showering routine on the morning of sampling	Sunscreens - Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss my face, Baby sunscreens that are “free” or “natural” Insect Repellents - Jason Natural Quit Bugging Me, Repel
	Lemon Eucalyptus Insect repellant, Herbal Armor, California
Sample Containers	
LDPE or glass containers and lined lids	HDPE or polypropylene
Teflon®-lined caps	Unlined polypropylene caps
Rain Events	
Waterproof or resistant rain gear	Gazebo tent that is only touched or moved prior to and following sampling activities
Equipment Decontamination	
Decon 90	Alconox® and/or Liquinox®
Water from an on-site well	Potable water from municipal drinking water supply
Food Considerations	
All food and drink, with exceptions noted on the right	Bottled water and hydration drinks (i.e. Gatorade® and Powerade®) to be brought and consumed only in the staging area

3.2.2 Equipment Cleaning Procedure

Field equipment that is utilized at each sample location will require cleaning between uses. Upon donning a new pair of nitrile gloves, equipment will be:

- Rinse with a Citranox® cleaning solution;
- Rinse with laboratory-provided, "PFAS-free" water; (Grade 3 distilled, Millipore deionized)
- Rinse with methanol; and,
- Rinse with laboratory-provided, "PFAS-free" water.

All rinsate should be collected in a sealed pail for disposal.

For groundwater sampling, the flow-through cell and any non-dedicated equipment (i.e. interface probe) that comes into contact with well water should be decontaminated between uses.

Field equipment used at locations that are suspected of containing AFFF (i.e. those that foam during shaking) will be cleaned as per above in triplicate.

SOP #4 – MONITORING WELL INSTALLATION

4.1 GENERAL DRILLING PROCEDURES

4.1.1 Drilling activities shall conform to applicable New York State Department of Environmental Conversation (NYSDEC) and shall be supervised by a qualified geologist, environmental scientist, or engineer. Well permits will not be required for the installation of temporary groundwater sampling points.

4.1.2 The location of borings shall be coordinated with SEDA before drilling commences. Utility location will be in accordance with SOP #1 – Site Reconnaissance, Preparation, and Restoration Procedures. Proposed well/boring locations will be adjusted if a location is not considered safe by utility locator services or by other methods (e.g. magnetometer).

4.1.3 Drilling shall be performed using a truck-mounted drill rig using direct push technology (DPT) techniques. The drill rig shall not leak any fluids that may enter the borehole or contaminate equipment placed in the hole. The use of rags or absorbent materials to absorb leaking fluids is unacceptable.

4.1.4 Vertical boreholes shall be straight and plumb. The position of the drill components at the start of drilling will be checked with a bubble level to ensure that boreholes begin in a plumb position. During drilling, factors that could affect the verticality of the borehole such as excessive speed of drilling or excessive down pressure on the drill string shall be noted and corrected.

4.1.5 A log of drilling activities shall be kept in a bound field notebook. Information in the log book shall include location, time on site, personnel and equipment present, down time, materials used, samples collected, measurements taken, and any other observations or information that would be necessary to reconstruct field activities at a later date. The drilling supervisor shall complete a daily drilling log at the end of each day of drilling.

4.2 MONITORING WELL CONSTRUCTION

Following completion of drilling, the well will be constructed using the following guidelines if the use of the GeoProbe SP-22 retractable screen sampler is determined not to be a viable option:

4.2.1 **Well Casings and Screen** - The well casings and screen will be 1-inch ID, flush joint, threaded, schedule 40 polyvinyl chloride (PVC) (Type 1, Grade 1 in accordance with ASTM D1784) with a threaded PVC plug. The screen sections will be ten feet in length with 0.01-inch openings (10-slot). The screen shall be free of any ink or printing.

Collection of Groundwater Samples for Perfluorooctanoic Acid (PFOA) and Perfluorinated Compounds (PFCs) from Monitoring Wells Sample Protocol

Samples collected using this protocol are intended to be analyzed for perfluorooctanoic acid (PFOA) and other perfluorinated compounds by Modified (Low Level) Test Method 537.

The sampling procedure used must be consistent with the NYSDEC March 1991 SAMPLING GUIDELINES AND PROTOCOLS http://www.dec.ny.gov/regulations/2636.html with the following materials limitations.

At this time acceptable materials for sampling include: stainless steel, high density polyethylene (HDPE) and polypropylene. Additional materials may be acceptable if proven not to contain PFCs. **NOTE: Grunfos pumps and bladder pumps are known to contain PFC materials (e.g. Teflon™ washers for Grunfos pumps and LDPE bladders for bladder pumps).** All sampling equipment components and sample containers should not come in contact with aluminum foil, low density polyethylene (LDPE), glass or polytetrafluoroethylene (PTFE, Teflon™) materials including sample bottle cap liners with a PTFE layer. Standard two step decontamination using detergent and clean water rinse should be considered for equipment that does come in contact with PFC materials. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFC materials must be avoided. Many food and drink packaging materials and "plumbers thread seal tape" contain PFCs.

All clothing worn by sampling personnel must have been laundered multiple times. The sampler must wear nitrile gloves while filling and sealing the sample bottles.

Pre-cleaned sample bottles with closures, coolers, ice, sample labels and a chain of custody form will be provided by the laboratory.

1. Fill two pre-cleaned 500 mL HDPE or polypropylene bottle with the sample.
2. Cap the bottles with an acceptable cap and liner closure system.
3. Label the sample bottles.
4. Fill out the chain of custody.
5. Place in a cooler maintained at 4 ± 2º Celsius.

Collect one equipment blank for every sample batch, not to exceed 20 samples.

Collect one field duplicate for every sample batch, not to exceed 20 samples.

Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, not to exceed 20 samples.

Request appropriate data deliverable (Category A or B) and an electronic data deliverable.

STANDARD OPERATING PROCEDURE 047
Per/Poly Fluorinated Alkyl Substances (PFAS) Field Sampling

1.0 Scope

Given the extremely low detection limits associated with PFAS analysis and the many potential sources of trace levels of PFAS, field personnel are advised to err on the side of caution by strictly following these protocols when sampling for PFAS to help mitigate the potential for false detections.

2.0 General Preparation and Considerations

Food considerations:

- Some food packaging has historically been treated with PFAS to improve its ability to resist wetting. As such, field personnel are to avoid the use of paper bags and not to bring food on site in any paper packaging (i.e. do not bring any fast food to the sites that uses and form of paper wrapping such as sandwiches, coffee in paper cups, etc.). If possible use cotton or hard plastic food containers. Avoid products such as aluminum foil, coated papers, and coated textiles.

- Foods that have been fried on a frying pan should not be brought to the site as the Teflon coating on most frying surfaces is made of a fluorinated coating and could represent a potential source of PFAS.

- Snacks and meals (lunch) are not to be eaten in the field vehicle or in the immediate vicinity of the monitoring wells (i.e. within 10m). When field personnel require a break to eat or drink, they should remove their gloves and coveralls and move to an appropriate location (preferably downwind). When finished, field personnel should then tidy up and put their coveralls and gloves back on prior to returning to the work area.

Field Gear:

- Water resistant, water proof or stain-treated clothing will not be worn during the field program. Field clothing to be worn on site should be restricted to natural fibers (preferably cotton) and not synthetic. Field clothing should be laundered with minimal use of soap, no fabric softener or scented products and after they have been cleaned, the clothing should be rinsed again with water only before drying (no fabric softener, etc.). Preferably, field gear should be cotton construction, old and well laundered. New cotton clothing may contain PFAS related treatments. The use of new clothing while sampling or sample handling shall be avoided. Gore-Tex™ consists of a PFAS membrane. Gore-Tex™ clothing shall not be worn during the sampling program.

- To avoid plastic coating or glue materials, waterproof field books are not to be used. Field reports should be on loose paper on Masonite or aluminum clip boards (i.e. plastic clip boards, binders or spiral hard cover notebooks are not acceptable) using a pencil. Pens and sharpies should not be used.

- Most safety footwear are made from leather and synthetic fibers that have been treated to provide some degree of waterproofing/increased durability and represent a source of trace PFAS. For the health and safety of field personnel, the protection for footwear must be maintained. As such, contact with safety footwear will take place after field personnel remove themselves from immediate vicinity of the monitoring well (i.e. 10m). Contact with footwear will not begin until new gloves are donned to minimize any potential for skin contact. Gloves will be disposed of when footwear contact is prior to beginning any other activities.

- Disposable nitrile gloves must be worn at all times. Further, a new pair of nitrile gloves shall be donned prior to the following activities at each sample location:

- Decontamination of re-usable sampling equipment;
- Prior to contact with sample bottles or "PFAS free" water containers
- Insertion of anything into the well (e.g. HDPE tubing, HydraSleeve, bailer, etc.);
- Insertion of silicon tubing into the peristaltic pump;
- Completion of monitor well purging, prior to sample collection;
- Handling of any QA/QC samples including field blanks and equipment blanks; and,
- After the handling of any non-dedicated sampling equipment, contact with non-decontaminated surfaces, or when judged necessary by field personnel.

Field Vehicle:

- The field vehicle seats may be treated with stain resistant products by the manufacturer. The seats of the vehicle shall be covered with a well laundered cotton blanket for the duration of the field program in order to avoid direct contact between field clothing and the seats of the vehicle.

Personnel Hygiene:

- Field personnel will not use shampoo, conditioner, body gel, cosmetic or hand cream as part of their personal cleaning/showering routine on the day of a sampling event, as these product may contain surfactants and represent a potential source of PFAS. It is strongly recommended that field personnel shower as per normal routine the night before and then rinse with water only on the morning of sampling event. Use of bar soap is considered acceptable, although soap containing moisturizing lotions should be avoided.

- Moisturizers, cosmetics and dental floss may contain PFAS and shall not be used throughout the duration of the field program, either on or off-site. Sunscreen and insect repellent also cannot be used.

- For washroom breaks, field personnel will remove themselves from the immediate vicinity of the sampling location (i.e. 10m) and then remove gloves and overalls. Field personnel should wash as normal with extra time for rinsing with water after soap use. When finished washing, the use of air dryer is preferred and the use of paper towel for drying is to be avoided (if possible).

Visitors:

- Visitors to the site are asked to remain at least 10m from sampling areas.

Rain Events:

- Field sampling will not take place when rain fall is consistent and persistent at a rate that it saturates the ground (i.e. formation of puddles) because rain gear is not permitted while sampling. Intermittent showers or fog are acceptable conditions to proceed. If/when showers occur; field gear will be removed from the monitoring well location until rain subsides.

- If project timelines are tight, teams should consider the use of a gazebo tent, which can be erected overtop of the monitoring well and provide shelter from the rain. It should be noted that the canopy material is likely a treated surface and should be treated as such; therefore, gloves should be worn when moving the tent, changed immediately afterwards and further contact with the tent should be avoided until all sampling activities have been finished and the team is ready to move on to the next site.

Table 1: Summary of Prohibited and Acceptable Items for Sampling of PFCs

PROHIBITED ITEMS	ACCEPTABLE ITEMS
Field Equipment	
Teflon® containing materials	High-density polyethylene (HDPE) materials
Low density polyethylene (LDPE)	Acetate liners
Aluminum foil	Silicon tubing
Waterproof field books	Loose paper (non-waterproof)
Plastic clipboards, binders, or spiral hard cover notebooks	Aluminum field clipboards or with Masonite
	Sharpies®, pens
Post-It Notes	
RE-usable Chemical (blue) ice packs	Regular ice in polyethylene bags (double bagged)
Field Clothing and Personal Protective Equipment (PPE)	
New cotton clothing or synthetic water resistant, waterproof, or stain- treated clothing, clothing containing Gore-Tex™	Well-laundered clothing, defined as clothing that has been washed 6 or more times after purchase, made of natural fibers (preferable cotton)
Clothing laundered using fabric softener	No fabric softener
Boots containing Gore-Tex™ Tyvek®	Boots made with polyurethane and polyvinyl chloride (PVC) Cotton Clothing
No cosmetics, moisturizers, hand cream, or other related products as part of personal cleaning/showering routine on the morning of sampling	Sunscreens - Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss my face, Baby sunscreens that are "free" or "natural" Insect Repellents - Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellant, Herbal Armor, California
Sample Containers	
LDPE or glass containers and lined lids	HDPE or polypropylene
Teflon®-lined caps	Unlined polypropylene caps
Rain Events	
Waterproof or resistant rain gear	Gazebo tent that is only touched or moved prior to and following sampling activities
Equipment Decontamination	
Decon 90	Alconox® and/or Liquinox®
Water from an on-site well	Potable water from municipal drinking water supply
Food Considerations	
All food and drink, with exceptions noted on the right	Bottled water and hydration drinks (i.e. Gatorade® and Powerade®) to be brought and consumed only in the staging area

3.0 Equipment Cleaning Procedure

Field equipment that is utilized at each sample location will require cleaning between uses. Upon donning a new pair of nitrile gloves, equipment will be:

- Rinse with a Citranox® cleaning solution;
- Rinse with laboratory-provided, "PFAS-free" water; (Grade 3 distilled, Millipore deionized)
- Rinse with methanol; and,
- Rinse with laboratory-provided, "PFAS-free" water.

All rinsate should be collected in a sealed pail for disposal.

For groundwater sampling, the flow-through cell and any non-dedicated equipment (i.e. interface probe) that comes into contact with well water should be decontaminated between uses.

Field equipment used at locations that are suspected of containing AFFF (i.e. those that foam during shaking) will be cleaned as per above in triplicate.

4.0 Borehole & Monitoring Well Sampling and Installation Procedures

- If a drill rig is being used to drill for soil cores or to install monitoring wells, clean nitrile gloves shall be worn prior to the collection of each continuous soil sample collection. Soil samples are collected in laboratory-supplied, "PFAS free" HDPE jars and labelled in pencil. Samples should be stored in coolers and kept at 0 – 4 ^{o}C until transported to the lab.

4.1 Well Condition Survey/Water Level Monitoring

- Under normal conditions, one of the first steps in conducting a groundwater sampling program is to complete a well condition survey (including the measurement of static water levels and monitor well depths). However, due to the extremely low detection limits for PFAS, the surveys should be conducted after groundwater purging and sampling to help mitigate the possibility of cross-contamination.

- Once all monitoring wells have been sampled, field personnel should conduct monitor well inspections and recorded water levels. An interface probe should be used to evaluate presence/absence of non-aqueous phase liquid (NAPL). Depth to water should be measured from the top of the PVC riser and the total depth of the well should also be measured. This information should be recorded on the Field Reports.

4.2 Monitoring Well Development and Purging

- Do not use Teflon or low density polyethylene tubing for purging or sample collection. High density polyethylene (HDPE) tubing is acceptable. No materials should be re-used between wells. Upon completion of use, all disposable materials (e.g. HDPE and/or silicon tubing) should be removed and placed in heavy duty garbage bags for disposal.

- During development of the well, sufficient energy should be created to agitate the water column and create flow reversals in the well screen, filter pack and formation to loosen fine-grained materials and draw them into the well. The pumping or bailing action should then draw all drilling fluids and fine-grained material out of the borehole and adjacent formation and then out of the well. Monitoring wells should be developed until visibly clear water is discharged from the well.

- Low flow purge and sampling techniques as per the US EPA "Low Stress (low flow) purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells" and the ASTM "Standard

Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations" should be followed.

- To purge the well, if using HDPE tubing and a peristaltic pump, the end of the tubing should be inserted to the approximate depth of the midpoint of the screened section of the monitor wells. The length of HDPE tubing to be inserted into each monitoring well will be measured and pre-cut to approximate lengths (i.e. previously measured arm span of field technician) to avoid contact with any materials other than the monitoring well and peristaltic pump. The tubing should be kept at least 0.6 m from the bottom of the well to prevent intake of particulates. Flow rates should be as low as can be reasonably achieved. Purge water should be collected and disposed of appropriately.

- Silicon tubing should direct the purge water through a flow-through cell for field parameter measurements of pH, conductivity, temperature, dissolved oxygen and turbidity. The instrument should be calibrated in the field prior to use, and the instrument and flow-through cell should be decontaminated at each monitor well location prior to purging.

- Field parameters should be recorded in intervals (generally of three minute duration) to ensure purge water has cycled through the flow-through cell. Wells should be sampled after field parameter measurements indicate stabilization, which allows collection of representative formation water (generally acceptable standards are three consecutive pH readings to within ±0.1 units, and three consecutive conductivity, temperature and dissolved oxygen measurements to within three percent). Turbidity is monitored, but should not be used as an indicator of purge completion. Field parameter measurements should be recorded at each well. As outlined below, drawdown should not be monitored throughout the purge in order to mitigate possible cross-contamination of groundwater samples introduced by the interface probe.

- If wells are suspected to be dewatering throughout the purge (i.e. reduced flow rate/difficulty pumping water or bubbles begin to come through the flow through cell), the pump should be turned off, and the water level allowed to recover for ½ hour, followed by sample collection. This should be documented in the Field Reports.

5.0 Sample Collection

5.1 General

Prior to sampling, confirm sample container composition (polypropylene versus HDPE) with the selected analytical laboratory. For each sample, the required minimum volume of surface water and groundwater is 250mL per USEPA 2009 [4] and the required minimum amount of soil or sediment is at least 2g on a dry weight basis per ASTM 2014. These sampling requirements may vary by laboratory. Prior to sampling, confirm sample size requirements with the selected analytical laboratory. Sampling volume is determined by the analytical laboratory and should be adapted to expected PFOS levels and analytical capacities. The instrumental limit of detection is the main factor limiting the sensitivity and the volume should be enough to reach quantification levels. [1]

For drinking water, each 250mL sample bottle *may be* required to contain a small amount (1.25g) of Trizma®, a buffering reagent that removes free chlorine from chlorinated drinking water (USEPA, 2009), or similar sample additive as specified by the selected analytical laboratory. Prior to sampling drinking water for PFAS analysis, confirm the need for additive with the selected analytical laboratory and the USACE chemist.

The use of chemical or gel-based coolant products (e.g.BlueIce®) to maintain samples at 4°C following sample collection is prohibited. The acceptable alternative is ice which has been double-bagged (polyethylene plastic) and secured to avoid meltwater from contacting sample containers during overnight or same-day delivery to the analytical laboratory.

Table 1 should be reviewed to identify other products that may contaminate the sampling processing area. If in doubt about a particular product or item in contact with environmental media to be sampled or in close proximity to operations, collect and analyze a rinsate sample using laboratory-supplied PFAS-free water. Filtration upon sample collection is not recommended since the filter may absorb PFASs or be a source of contamination. (1)

Support personnel that are within 2–3m of the processing area are considered subject to the same restrictions related to precautionary measures for clothing and food, as applied to sampling personnel. During sample processing and storage, minimize the exposure of the sample to light.

5.2 Sample Containers:

- Different laboratories may supply sample collection containers of varying sizes dependent on the type of media to be sampled (i.e. soil, groundwater, etc.).

- Results of ESG's inter-laboratory comparison suggest that standardizing sample container size and performing whole-sample analysis (i.e. no sub-sampling) will optimize inter-laboratory comparability and ensure result consistency for departments, regardless of which laboratory is used.

- The use of different laboratory testing methods (i.e., SPE and DI) could be responsible for result variability between laboratories. Sub-sampling is inherent to the direct injection method and introduces possible bias in either direction. To achieve "reportable quantitative results" in aqueous samples, the use of the SPE method is recommended.

- All samples should be collected in polypropylene or high density polyethylene (HDPE) bottles fitted with an unlined (no Teflon), polypropylene screw cap.

- Container Labels will be completed using pen/pencil (i.e. NO MARKERS) after the caps have been placed back on each bottle.

- Glass containers should also be avoided due to potential loss of analyte through adsorption.
  Sample Shipping Requirements:

- Samples should be maintained between 0–4 ºC during shipping. Samples should be shipped via courier service with priority overnight delivery. Tracking numbers for all shipments should be provided once they have been sent out so to ensure their timely delivery.

5.3 Soil Sampling

- Clean nitrile gloves shall be worn prior to the collection of each sample.
- Samples are to be collected using a stainless steel trowel.
- Soil samples are collected in laboratory-supplied, "PFAS free" HDPE jars and labelled in pencil. Samples should then be placed in coolers and kept at between 0 – 4 ºC until transported to the lab.
- Soil and sediment core samples must be collected directly from single-use PVC liners that must not be decontaminated or reused at different locations.

5.4 Groundwater Sampling

- Sample collection will take place upon stabilization of field parameters. The silicon tubing should be disconnected from the flow-through cell, enabling collection of groundwater samples prior to passing through the cell.

- Prior to collection of samples, field personnel must wash their hands and don a new set of nitrile gloves. Gloved hands must not be used to subsequently handle papers, pens, clothes, etc., prior to the collection of PFAS samples. The PFAS samples bottle caps must remain on the bottle until immediately prior to sample collection and the bottle immediately sealed after sample collection. This will minimize potential loss of PFAS, through volatilization. The bottle cap must remain in the other hand of the sampler, until replaced on the bottle. PFAS sample bottles will not be rinsed during sampling.

- During PFAS sampling, water turbulence should be minimized to avoid potential volatilization from aqueous solution; this could include: adjusting pump discharge prior to sampling and inclining the sample bottle neck, during filling of the bottle. Ensure the rim of the bottle does not come into direct contact with the pumping equipment or tubing. Environment Canada states that while PFOS has low volatility, many of its precursors are volatile.

- Groundwater samples will be collected in pre-labelled, laboratory-supplied "PFAS free" HDPE sample vials. Each "PFAS clean" sample vial should then be placed in a sealed Ziploc© bag. Labelling information and time of sampling should be recorded on the Field Reports. All sampling materials should be treated as single use and disposed following completion of sampling at each monitor well.

- A small portion of the sample (~10-25mL) should be collected and shaken by the sample collector on site. If foaming is noted within the sample, this should be documented when samples are submitted for analysis; the 'shaker test' vial can then be disposed of. This shaker test provides information about how each of the samples should be handled analytically.

- Samples should be placed in coolers and kept at a cool temperature until transportation to the lab. Samples must be kept at between 0–4 ºC.

5.5 Sediment Sampling

- Sediment samples should be either collected manually using a stainless steel trowel or collected using petite ponar grab sampler depending on field condition at each sampling location during sampling program.

- Sediment samples will be collected within the upper 10 cm of sediment.

- For a sample to be acceptable the overlying water is present and has low turbidity; the sampler is not overfilled or contains vegetation; the sediment is undisturbed and sampler shows no signs of winnowing or leaking.

- Overlying water will be decanted and a stainless steel trowel will be used to collect only the upper 5 cm of sediment.

- Sediment will be placed directly into laboratory supplied containers suitable in both material and size, placed in supplied packaging and immediately stored in cooler with ice to ensure a whole sample is collected to mitigate issues with sample collection. Jar lids will remain in the gloved hand of the sampler until replaced on the bottle. Labels will be completed using pen/pencil (i.e. NO MARKERS) after the caps have been placed back on each bottle.

5.6 Surface Water Sampling

- Where surface water samples and sediment samples are collected at the same location, surface water samples will be collected first to minimize the siltation.

- Surface water must be collected by inserting a capped sampling container (polypropylene or HDPE) with

the opening pointing down to avoid the collection of surface films. At the time of container opening, the container must be more than 10cm from the sediment bed and more than 10cm below the surface water level and as close to the center of the channel as possible, where practicable. Point the container up to fill so that gloved hands, sample container, and sampler are downstream of where sample is being collected.

- Surface water pH, conductivity, temperature and total dissolved solids (TDS) will be measured at each location after sediment sampling.

- Surface water samples will be placed directly into laboratory supplied containers suitable in both material and size, placed in supplied packaging and immediately stored in cooler with ice to ensure a whole sample is collected to mitigate issues with sample collection. Jar lids will remain in the gloved hand of the sampler until replaced on the bottle. Labels will be completed using pen/pencil (i.e. NO MARKERS) after the caps have been placed back on each bottle.

- Sample collection will take place prior to measurement of water level and well depth as to minimize the potential for cross-contamination due to surface water/sediment contact with field equipment.

6.0 Quality Assurance & Quality Control Sampling

6.1 Equipment Blanks

- QA/QC sampling will include daily collection of equipment blanks using the laboratory supplied 'PFAS free' water. Sample will be collected in laboratory supplied containers. Bottle caps will remain in the hand of the sampler until replaced on the bottle. Labels will be completed using pen/pencil (i.e., **NO MARKERS**) after the caps have been placed back on each bottle. Samples will be sealed in suitable packaging and stored on ice in a cooler for shipment to the lab.

  For peristaltic pump tubing, laboratory supplied "PFAS free" water should be poured into a clean HDPE sample bottle and then pumped through new HDPE tubing using the peristaltic pump (with new silicon tubing).

6.2 Field Duplicates

- QA/QC sampling will include the collection of one blind field duplicate for each 10-sample group. These samples will be collected immediately after the initial samples into laboratory supplied "PFAS clean" sample containers. Bottle caps will remain in the hand of the sampler until replaced on the bottle. Labels will be completed using pen/pencil (i.e., **NO MARKERS**) after the caps have been placed back on each bottle. Samples will be sealed in suitable packaging and stored on ice in a cooler for shipment to the lab.

6.3 Field Blank

- QA/QC sampling will include daily submission of laboratory supplied field blanks. The samples will be brought to the site in the laboratory supplied sample bottles and opened during the collection of a sample and then released. Bottle caps will remain in the hand of the sampler until replaced on the bottle. Labels will be completed using pen/pencil (i.e., **NO MARKERS**) after the caps have been placed back on each bottle. Samples will be sealed in suitable packaging and stored on ice in a cooler for shipment to the lab.

6.4 Trip Blank

- QA/QC sampling will include the submission of one laboratory supplied trip blank. The sample will be brought to the site in laboratory supplied sample bottle and will remain inside the cooler during the sampling program. Labels will be completed using pen/pencil (i.e., **NO MARKERS**). Samples will be sealed in suitable packaging and stored on ice in a cooler for shipment to the lab.

6.5 Spiked Samples – Laboratory Performance Evaluation

- QA/QC sampling will include the submission of two laboratory supplied spiked samples of known concentration (one low level and one moderate level based on previous PFAS sample results) submitted blindly by the environmental consultant. These samples will be utilized in lieu of certified reference material (CRM) to assist in data validation. Labels will be completed using pen/pencil (i.e., **NO MARKERS**). Samples will be sealed in suitable packaging and stored on ice in a cooler for shipment to the lab.

6.6 Laboratory Analytical QA/QC

- Internal laboratory QA/QC should consist of one laboratory blank per batch of samples; one soil or water matrix spikes and generally isotopically-labelled surrogates for analysis. Labels will be completed using pen/pencil (i.e., **NO MARKERS**). Samples will be sealed in suitable packaging and stored on ice in a cooler for shipment to the lab.

- As part of the internal QA/QC, relative percent difference (RPD) should be calculated between samples and corresponding field or laboratory duplicates. The laboratory quality assurance portion of the laboratory certificates should be reviewed to verify that all calculations/recoveries were within acceptable limits as established by the laboratory method.

6.7 Laboratory Analytical Sub-sampling

The results of ESG's inter-laboratory comparison, however, still imply that subsampling is a significant cause of inter-laboratory variability, and it is a practice inherent to the DI method. The Ontario Ministry of the Environment recommends use of the SPE method to achieve reportable quantitative results for drinking water samples.

7.0 Laboratories

An accredited laboratory for analysis of PFAS will be used.

References:

(1) United Nations Environment Programme (UNEP), Division of Technology, Industry and Economics. 2015. *PFAS analysis in water for the Global Monitoring Plan of the Stolkholm Convention, Set-up and guidelines for monitoring.* April.

(2) Government of Western Australia, Department of Environmental Regulation. 2016. *Interim Guideline on the Assessment and Management of Perfluoroalkyl Substances (PFAS), Contaminated Sites Guidelines.* February.

(3) Environmental Fate and Effects of Poly-and Perfluoroalkyl Substances (PFAS). 2016. CONCAWE (European Industrial Consortium)

(4) USEPA Method 537: Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS), version 1.1, September 2009. National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency.

Appendix E

Historical Reports

The following historical reports are provided on the electronic (CD) version of this report.

Ash Landfill Round 21 (June 2016) Long-Term Monitoring Report

2016 OB Grounds Round 11 Long-Term Monitoring Report

2016 SEAD-25 Year 9 Long Term Monitoring Report

2015 SEAD 16/17 Year 8 Long Term Monitoring Report

SEAD-26 Site Map and Boring Logs

SEAD-122E Site Map and Boring Logs

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100 High Street, 4th Floor • Boston, Massachusetts 02110 • (617) 946-9400 • Fax (617) 946-9777 • www.parsons.com

TECHNICAL MEMORANDUM

Date: November 09, 2016

To: Julio Vazquez, USEPA
Melissa Sweet, NYSDEC
Mark Sergott, NYSDOH

From: Beth Badik, Parsons

Subject: Round 21 – Long-Term Monitoring Results for the Ash Landfill at Seneca Army Depot Activity, Romulus, New York

1. INTRODUCTION

Groundwater monitoring is being performed as a requirement of post-closure remedial action operations for the Ash Landfill Operable Unit (OU) at the Seneca Army Depot Activity (SEDA or the Depot), Seneca County, New York. Three sets of dual biowalls were installed at the Ash Landfill in September and October 2006 to treat the chlorinated ethenes plume. In accordance with the Post-Closure Monitoring and Maintenance Plan (PCMMP) presented in the "Remedial Design Report for the Ash Landfill Operable Unit, Revised Final" (RDR) (Parsons, 2006), quarterly groundwater monitoring was required during the first year of biowall operation to monitor the plume and the biowall treatment process.

In the "Annual Report and One-Year Review for the Ash Landfill" (Parsons, 2008), the Army recommended that the frequency of long-term groundwater monitoring (LTM) events at the Ash Landfill OU be reduced from quarterly to semi-annually. This recommendation was approved by the U.S. Environmental Protection Agency (USEPA) and the New York Department of Environmental Conservation (NYSDEC). A summary of the LTM events are presented below:

Exhibit 1.1 – LTM Events Summary

Round #	Round ID	Year ID	Date
1	1Q2007	Year 1	January 2 through January 4, 2007
2	2Q2007	Year 1	March 15 through March 17, 2007
3	3Q2007	Year 1	June 5 through June 6, 2007
4	4Q2007	Year 1	November 13 through November 16, 2007
5	5R2008	Year 2	June 24 through June 26, 2008
6	6R2008	Year 2	December 11 through December 15, 2008
7	7R2009	Year 3	June 2 through June 4, 2009
8	8R2009	Year 3	December 14 through December 18, 2009
9	9R2010	Year 4	June 28 through July 2, 2010
10	10R2010	Year 4	December 14 through December 19, 2010
11	11R2011	Year 5	July 18 through July 22, 2011

Exhibit 1.1 – LTM Events Summary

Round #	Round ID	Year ID	Date
12	12R2011	Year 5	December 12 through December 15, 2011
13	13R2012	Year 6	June 18 through June 22, 2012
14	14R2012	Year 6	December 10 through December 14, 2012
15	15R2013	Year 7	July 8 through July 12, 2013
16	16R2013	Year 7	December 9 through December 14, 2013
17	17R2014	Year 8	June 17 through June 22, 2014
18	18R2014	Year 8	December 15 through December 19, 2014
19	19R2015	Year 9	June 2 through June 6, 2015
20	20R2015	Year 9	December 15 through December 19, 2015
21	21R2016	Year 10	June 14 through June 17, 2016

This letter report presents the results of the first round of Year 10 semi-annual monitoring, referred to as Round 21 (21R2016), which was completed between the 14th and 17th of June 2016.

1.1 Objective

LTM is required at the Ash Landfill OU because contaminant concentrations in the groundwater exceeded the NYSDEC Ambient Water Quality Criteria Class GA standards for groundwater. Two types of LTM are being performed: (i) plume performance monitoring and (ii) biowall process monitoring.

Performance monitoring is conducted to measure groundwater contaminant concentrations and the effectiveness of the biowalls as a remedy for the Ash Landfill OU. The LTM results from performance monitoring are used to demonstrate that contaminants of concern (COCs) do not exceed groundwater standards at the off-site sentinel well, MW-56. Ultimately, these data will be used to document that concentrations of COCs meet the Class GA groundwater standards on-site before LTM is discontinued.

The second type of monitoring, biowall process monitoring, is conducted at select wells located either within or immediately downgradient of the biowalls. Biowall process monitoring is intended to assess if and when the biowalls may require additional substrate (e.g., injection of emulsified vegetable oil).

1.2 Site Description

SEDA is a 10,587-acre former military facility located in Seneca County in the Towns of Varick and Romulus, New York. SEDA is located between Seneca Lake and Cayuga Lake and is bordered by New York State Highway 96 on the east, New York State Highway 96A on the west, and sparsely populated farmland on the north and south. The Depot was owned by the United States Government and operated by the Department of the Army between 1941 and 2000, when its military mission ceased. Since 2000, more than 8,250 acres of the facility have been transferred to other parties for alternate uses.

The Ash Landfill OU is comprised of five historic Solid Waste Management Units (SWMUs): the Incinerator Cooling Water Pond (SEAD-3), the Ash Landfill (SEAD-6), the Non-Combustible Fill Landfill (NCFL) (SEAD-8), the Refuse Burning Pits (SEAD-14), and the former Abandoned Solid Waste

Incinerator Building (SEAD-15) (**Figure 1**). The Debris Piles were located near SEAD-14. The Ash Landfill (SEAD-6) is the source of a groundwater plume emanating from the western side of the landfill. The groundwater plume extends 1,100 feet from the original source area to the western property line of the Depot. The plume consists of chlorinated ethenes, primarily trichloroethene (TCE), and the dichloroethene isomer cis-1,2-dichloroethene (cDCE).

1.3 Background

The Ash Landfill OU is underlain by a broad north-to-south trending series of rock terraces covered by a mantle of glacial till. As part of the Appalachian Plateau, the region is underlain by a tectonically undisturbed sequence of Paleozoic rocks consisting of shales, sandstones, conglomerates, limestones, and dolostones. At the Ash Landfill site, these rocks (the Ludlowville Formation) are characterized by gray, calcareous shales and mudstones, and a thin layer of limestone, with numerous zones of abundant invertebrate fossils. Locally, the shale is soft, gray, and fissile. The shale, which has a thin weathered zone at the top, is overlain by 2 to 3 feet of Pleistocene-age[1] till deposits. The till matrix varies locally, but generally consists of unsorted silt, clay, sand, and gravel. At the Ash Landfill OU, the thickness of the till generally ranges from 4 to 15 feet. At the location of the biowalls, the thickness of the till and weathered shale is approximately 10 to 15 feet (Brett et al., 1995).

Groundwater is present in both the shallow till/weathered shale layer and in the deeper competent shale layer. In both water-bearing units, the predominant direction of groundwater flow is to the west, toward Seneca Lake. Based on historical data, the wells at the Ash Landfill OU exhibit rhythmic and seasonal fluctuations in the water table and the saturated thickness. Historic data at the Ash Landfill OU indicate that the saturated interval is thinnest (generally, between 1 and 3 feet thick) in the month of September and thickest (generally, between 6 and 8.5 feet thick) between December and March (Parsons Engineering Science Inc., 1994).

The average linear velocity of the groundwater in the till/weathered shale layer was calculated during the Remedial Investigation (RI) in 2004 using the following parameters: 1) average hydraulic conductivity of 4.5×10^{-4} centimeters per second (cm/sec), or 1.28 feet per day (ft/day), 2) estimated effective porosity of 15% to 20% (0.15 - 0.20), and 3) groundwater gradient of 1.95×10^{-2} feet per foot (ft/ft) (Parsons Engineering Science, Inc., 1994). The average linear velocity was calculated as 0.166 ft/day or 60.7 feet per year (ft/yr) at 15% effective porosity and 0.125 ft/day or 45.5 ft/yr at 20% effective porosity. The actual velocity of on-site groundwater may be locally influenced by zones of higher-than-average permeability; these zones are possibly associated with variations in the actual porosity of the till/weathered shale layer.

Three dual biowall systems, known as A1/A2, B1/B2, and C1/C2, were constructed at the Ash Landfill OU in September 2006. The purpose of the dual biowall systems is to address chlorinated solvent contamination in groundwater. Biowalls A1/A2, B1/B2, and C1/C2 were constructed perpendicular to the flow of the chlorinated solvent plume in the locations prescribed in the RDR. Each biowall system was intended to be

[1] The Pleistocene Age occurred 2,588,000 to 11,700 years ago years before present.

comprised of separate, paired parallel trenches filled with organic substrate. However, the entire length of Biowalls A1/A2 and the northern portion of B1/B2 were constructed as a single double-width trench (minimum of 6 feet in width) due to unstable soil conditions that caused trench widening. Approximately 2,705 linear feet (lf) of biowall were constructed in the areas downgradient of the Ash Landfill. The biowalls range in depth from 7 feet below ground surface (bgs) to 18.5 feet bgs. Each trench was filled with an organic substrate (mulch/sand mixture coated with vegetable oil) that enhances biodegradation of chlorinated solvents. Details of the final biowall system are documented in the "Draft Construction Completion Report for the Ash Landfill OU" (Parsons, 2007).

TCE and the dichloroethene isomer cDCE are the most prevalent chlorinated ethenes found in groundwater at the Ash Landfill. The TCE plume originates from the Ash Landfill (SEAD-6) and extends westward approximately 1,100 feet to the western boundary of the Depot. The areal extent of TCE in the groundwater in January 2000 is illustrated in **Figure 2**. Subsequent monitoring has shown little change in the plume's areal extent since then. Sampling indicates that the TCE plume has not reached monitoring well (MW-56) located on the adjacent property.

1.4 Description of Technology Used in Biowalls

Reductive dechlorination is the most important process for natural biodegradation of highly chlorinated solvents (USEPA, 1998) (**Figure 3)**. Complete dechlorination of TCE and other chlorinated solvents is the goal of anaerobic biodegradation with mulch biowall technology.

Biodegradation causes measurable changes in groundwater geochemistry. These changes in groundwater chemistry can be used to evaluate the effectiveness of substrate addition in stimulating biodegradation. For anaerobic reductive dechlorination to be an effective process, generally groundwater must be sulfate-reducing or methanogenic. Thus, groundwater in which anaerobic reductive dechlorination is occurring should have the following geochemical signature:

- Depleted concentrations of dissolved oxygen (DO), nitrate, and sulfate;
- Elevated concentrations of manganese, ferrous iron, methane, carbon dioxide, chloride, and alkalinity; and
- Reduced oxidation-reduction potential (ORP).

Anaerobic reductive dechlorination relies on the flow of groundwater, under a natural hydraulic gradient, through the biowall to promote contact with slowly-soluble organic matter. As the groundwater flows through the organic matter in the biowall, an anaerobic treatment zone is established in the biowall. A treatment zone may also be established downgradient of the biowall as the organic matter migrates with the groundwater and stimulates microbial processes.

Solid-phase organic substrate used to stimulate anaerobic biodegradation of chlorinated ethenes includes plant mulch and compost. To provide active microbial populations for degradation of the substrate in the subsurface, mulch may be composted prior to emplacement or mulch may be mixed with an outside source of compost. Mulch is primarily composed of cellulose and lignin and contains "green" plant material that

provides nitrogen and nutrients for microbial growth. These substrates are mixed with coarse sand and placed in a trench or excavation in a permeable reactive biowall configuration. Biodegradable vegetable oil may also be added to the mulch mixture to increase the availability of soluble organic matter.

Degradation of the organic substrate by microbial processes in the subsurface provides a number of breakdown products, including metabolic acids (e.g., butyric and acetic acids). The breakdown products and acids produced by degradation of mulch in a saturated subsurface environment provide secondary fermentable substrates for the generation of hydrogen, which is the primary electron donor utilized in anaerobic reductive dechlorination of chlorinated ethenes. Thus, a mulch biowall has the potential to stimulate reductive dechlorination of chlorinated ethenes for many years. If necessary, mulch biowalls can be recharged with liquid substrates (e.g., emulsified vegetable oil) to extend the life of the biowall. Vegetable oil is a substrate that is readily available to microorganisms as a carbon source that helps establish and continually develop the microbial population. Used in combination with mulch, vegetable oil has the potential to enhance and extend the duration of organic carbon release.

2. GROUNDWATER MONITORING ACTIVITIES

All of the Ash Landfill groundwater samples were collected using low-flow sampling techniques. Sampling procedures, sample handling and custody, holding times, and collection of field parameters were conducted in accordance with the "Final Sampling and Analysis Plan for Seneca Army Depot Activity (SAP)" (Parsons, 2005).

During Round 21 of groundwater sampling, all 14 monitoring wells were sampled between the 14th and 17th of June 2016. The monitoring wells at the Ash Landfill OU are classified into three groups: on-site plume performance monitoring wells, biowall process monitoring wells, and off-site performance monitoring wells. The wells in each group are listed in **Table 1**.

The list of five biowall process monitoring wells includes three wells from the plume performance group (MWT-23, MWT-28, and MWT-29). These three wells are either within or immediately downgradient of the biowalls and are used to assess if and when the biowalls may require additional substrate.

"Annual Report - Year 1" (Parsons, 2008) recommended that groundwater samples collected from monitoring wells PT-17 and MWT-7, which are on-site plume performance wells, be analyzed for the geochemical parameters that are included for the process monitoring wells, in order to better monitor the progress of the treatment zone.

During Round 21, groundwater samples were collected at the wells listed in **Table 1**. Samples from wells in the biowall process monitoring group (MWT-23, MWT-26, MWT-27, MWT-28, and MWT-29) and from two wells from the on-site plume performance group (PT-17 and MWT-7) were submitted to TestAmerica Laboratories, Inc. in Savannah, Georgia to be analyzed for:

- Volatile Organic Compounds (VOCs) by USEPA SW846 Method 8260B
- Total organic carbon (TOC) by USEPA SW846 Method 9060A
- Sulfate by USEPA Method 300.1

Samples from these wells were also submitted to Pace Analytical located in Pittsburgh, Pennsylvania for analysis for methane, ethane, and ethene (MEE) by Method RSK 175. The remaining wells in the on-site plume performance group (PT-18A, PT-22, PT-24, MWT-22, MTW-24, and MTW-25) and the off-site performance monitoring well (MW-56) were analyzed for VOCs by USEPA SW846 Method 8260B, only.

The TestAmerica Savannah, GA and the Pace Analytical laboratories are certified by the Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) and the NELAC National Environmental Laboratory Accreditation Program (NELAP) for the above analyses/analytical methods for both potable and non-potable water.

During sampling in the field, the following geochemical parameters were recorded for the duration of low-flow sampling for each groundwater sample:

- pH, ORP, and conductivity were measured with a Horiba U-52 multi-parameter instrument;
- DO and temperature were measured with a YSI 85 meter; and
- Turbidity was measured with a LaMotte 2020 or similar turbidity meter.

In addition, a HACH® DR/850 Colorimeter was used in the field to measure manganese and ferrous iron at PT-17, MWT-7, MWT-23, MWT-26, MWT-27, MTW-28, and MWT-29. Manganese and ferrous iron were measured by USEPA Method 8034 and USEPA Method 8146, respectively. A summary of the samples collected is presented in **Table 1**.

3. GROUNDWATER MONITORING RESULTS

Groundwater Elevations

Groundwater levels were collected during the Round 21 sampling event on June 14, 2016. Historic groundwater elevations and groundwater elevations from the most recent round are presented in **Table 2**. Groundwater contours based on the most recent groundwater elevations measured in June 2016 are shown in **Figure 4**. Historically, groundwater elevation measurements indicate that groundwater generally flows west across the Ash Landfill OU site, perpendicular to the biowalls. The groundwater elevation trends are plotted on **Figure 5**. Groundwater levels measured during the 21R2016 sampling event are consistent with historical summer groundwater levels (**Figure 5**).

Geochemical Results

Biodegradation causes measurable changes in groundwater geochemistry that can be used to evaluate the effectiveness of substrate addition in stimulating biodegradation. For anaerobic reductive dechlorination to be an effective process, typically groundwater will be sulfate-reducing or methanogenic. As mentioned above, geochemical parameters measured in the field that serve as water quality indicators (i.e., pH, ORP, conductivity, DO, and temperature) were recorded for all wells in the LTM program. Analysis for the additional geochemical parameters of TOC, sulfate, and methane/ethene/ethane and field tests for ferrous iron and manganese were completed at PT-17, MWT-7, MWT-23, MWT-26, MWT-27, MTW-28, and MWT-29 (**Table 1**). Analysis of these geochemical parameters indicates that conditions are conducive for anaerobic reductive dechlorination to occur if the following geochemical signatures are identified:

- Depleted concentrations of DO and sulfate;
- Elevated concentrations of methane;
- Reduced ORP;
- Elevated concentrations of soluble organic substrate in groundwater (TOC); and
- Relative increase in the concentrations of ferrous iron and manganese.

Geochemical parameter results are shown in **Table 3**. This table is organized with the most upgradient well listed first and the most downgradient well listed last. A comparison of the geochemical parameters for wells MWT-26 (upgradient of Biowall B1) to MWT-28 (in Biowall B2), summarized below, demonstrates the change in geochemistry across the B1/B2 Biowall.

***Dissolved Oxygen*.** Dissolved oxygen is the most favored electron acceptor used by microbes for the biodegradation of organic carbon, and its presence can inhibit the biodegradation of chlorinated ethenes. A typical pattern of DO levels, from upgradient to downgradient across a biowall, would demonstrate the depletion of DO (e.g., < 0.5 mg/L) within the biowall relative to a location immediately upgradient of the biowall.

- The DO level at the well upgradient of Biowall B1 (MWT-26) was 0.23 mg/L.
- Within Biowalls B1/B2 (MWT-27 and MWT-28), DO levels are depleted with concentrations of 0.13 mg/L and 0.48 mg/L, respectively.
- Depleted levels of DO were observed in well MWT-23 (Biowall C2) with a concentration of 0.13 mg/L.

Typical low levels of DO (<1 mg/L) in the biowalls compared to the upgradient well location indicates that DO is depleted due to the biological activity encouraged by the biowall substrate (**Table 3**). Low levels of DO in the well between the biowalls suggest that the environment between the biowalls is anaerobic. The depletion of DO enhances the potential for degradation of chlorinated ethenes in groundwater.

Sulfate. Sulfate is used as an electron acceptor during sulfate reduction, competing with anaerobic reductive dechlorination for available substrate/electron donor. Sulfate levels lower than 20 mg/L are desired to prevent inhibition of reductive dechlorination of chlorinated ethenes (USEPA, 1998).

- The sulfate level at MWT-26 (upgradient of Biowalls B1/B2) was 590 mg/L.
- The sulfate concentrations at MWT-27 (in Biowall B1), MWT-28 (in Biowall B2), and MWT-23 (in Biowall C2) were 11 mg/L, 0.77 J mg/L, and 5.9 mg/L, respectively, which were in the desired range to support reductive dechlorination.

The sulfate levels detected within the biowalls were two orders of magnitude lower than the concentration of sulfate detected upgradient of Biowalls B1/B2 at MWT-26 (**Table 3**). These conditions indicate that sulfate is also being depleted by anaerobic microorganisms, and anaerobic dechlorination within the biowalls is active.

Methane. The presence of methane in groundwater is indicative of strongly reducing methanogenic conditions. Elevated concentrations of methane are an indication that reducing conditions are optimal for anaerobic reductive dechlorination to occur. In the biowalls, the concentrations of methane increased by two orders of magnitude (**Table 3**):

- Upgradient well (MWT-26): 170 µg/L.
- Biowall B1 (MWT-27): 17,000 µg/L (average of sample and associated duplicate)
- Biowall B2 (MWT-28): 15,000 µg/L
- Biowall C2 (MWT-23): 14,000 µg/L

These data demonstrate that there is an increase in the level of methanogenic activity within the biowalls relative to upgradient locations.

Oxidation-Reduction Potential. ORP indicates the level of electron activity in groundwater and the tendency of groundwater to accept or transfer electrons. Low ORP, less than 50 millivolts (mV using Ag/AgCl as the reference electrode), is typically considered the upper limit below which reductive dechlorination is possible, and even lower values of less than -100 mV suggests an increased likelihood for anaerobic reductive dechlorination to occur (USEPA, 1998). During Round 21, ORP readings were:

- Upgradient well (MWT-26): 77 mV
- Biowall B1 (MWT-27): -79 mV
- Biowall B2 (MWT-28): -70 mV
- Biowall C2 (MWT-23): -64 mV

The ORP levels within the biowalls are all negative and less than the benchmark of 50 mV, and significantly lower than the ORP level at the upgradient well, which suggests that the biowalls would likely support significant sulfate reduction, methanogenesis, and anaerobic reductive dechlorination **(Table 3)**. Historically, the ORP levels in the biowalls were closer to the preferred reference level of -100 mV; however, the ORP levels in all of the biowalls are well below the 50 mV reference level where a reductive pathway is possible (USEPA, 1998).

Total Organic Carbon. The presence of organic substrate is necessary to fuel anaerobic degradation processes. In reductive dechlorination, carbon acts as an energy source for anaerobic bacteria and drives reductive dechlorination. Concentrations of TOC greater than 20 mg/L are sufficient to maintain sulfate reducing and methanogenic conditions (USEPA, 1998). TOC concentrations in Biowalls B1/B2 were greater than the TOC concentrations upgradient of the biowalls (**Table 3**). The concentrations of TOC in the biowalls and upgradient well are as follows (**Table 3**):

- Upgradient well (MWT-26): 5.0 mg/L
- Biowall B1 (MWT-27): 26.5 mg/L (average of sample and associated duplicate)

- Biowall B2 (MWT-28): 16 mg/L
- Biowall C2 (MWT-23): 4.7 mg/L

The TOC concentrations at Biowalls B1/B2 were greater than the TOC concentration at the upgradient well and, historically greater than, the reference level of 20 mg/L. As the biowalls age, there is a decrease in the concentration of TOC as readily degraded organics (i.e., vegetable oil and cellulose) in the mulch mixture are consumed. This is evident as TOC concentrations are observed to decrease over the timespan of the LTM and the TOC concentration in Biowall B2 has dropped below the reference level (Table 3). Although TOC concentrations are decreasing, the concentrations within the B1/B2 biowalls remain sufficiently high enough to serve as an energy source for anaerobic bacteria and that the change in TOC concentrations appears to have little impact on the efficiency at which chlorinated ethenes are degraded within the biowalls. The TOC concentration in Biowall C2 (MWT-23) is below the reference level for anaerobic biodegradation processes and is about equal to the value of the upgradient well. There is no specific value for TOC which would indicate that the environment within Biowall C2 is no longer adequate for the reduction of VOC concentrations, therefore the geochemical conditions within the biowalls will continue to be monitored in future rounds.

Chemical Results

Table 4 reports the concentrations of chlorinated ethenes detected in groundwater during the first year of quarterly monitoring (Quarters 1-4) and subsequent semi-annual monitoring events (Rounds 5-21). The most recent round of groundwater sampling was performed approximately 10 years and 8 months after installation of the biowalls. The primary contaminants detected at the site include TCE, cDCE, and vinyl chloride (VC). A summary of the detected VOCs from the current sampling round is presented in **Table 5**. Historical concentrations of TCE, cDCE, and VC and their sampling location are summarized on a site figure (**Figure 2**). In the current round of sampling, TCE was detected in 9 of the 14 wells; TCE was not detected in Biowall B1 (MWT-27), B2 (MWT-28) or Biowall C2 (MWT-23).

A downgradient profile showing the concentration of TCE, and its degradation products (cDCE and VC), with distance from well PT-18A illustrates the dechlorination of TCE in the biowalls and the production of its associated degradation products (**Figure 6**). The concentrations of TCE and its daughter products (cDCE and VC), as well as the correlation between TCE degradation and the production of the daughter products, are evaluated below.

TCE TCE was detected above its Class GA Standard (5 µg/L) in five wells (PT-18A, MWT-25, PT-22, PT-17, and MWT-7) with a maximum of 280 µg/L at PT-18A (**Table 4**). TCE was detected upgradient of the biowall system at concentrations of 6.9 µg/L (MWT-25) and 2.1 µg/L (MWT-26), respectively. A plot of TCE concentrations versus distance from the source area illustrates that TCE is reduced to concentrations close to or below the detection limit as it flows through the A1/A2 and B1/B2 biowalls (**Figure 6**). The concentration of TCE rebounds with increasing distance from the biowalls as groundwater transitions outside the treatment zone. Geochemical parameters at PT-17 and MWT-7 (i.e., ORP and ferrous iron) indicate that conditions at this point in the plume are not as supportive of anaerobic degradation as the

conditions further upgradient in the plume (**Table 3**). TCE was not detected at the off-site sentinel well (MW-56).

Cis-1,2-DCE Similar to the reduction in TCE concentration detailed above, the concentration of cDCE is similarly reduced as cDCE enters the biowalls (**Figure 6**). As expected due to the reduction of TCE and production of its degradation products, concentrations of cDCE subsequently rebound downgradient of the biowalls (**Figure 6** and **Table 4**). The increase in concentration of cDCE observed immediately downgradient of the B1/B2 and C2 biowalls may be an indication that TCE is being converted to cDCE, the next sequential step in the dechlorination process. This suggests that the treatment zone in the biowalls is being sustained. cDCE concentrations in the biowalls were measured below the Class GA groundwater standard (5 µg/L). cDCE was detected at a concentration below the Class GA groundwater standard at the off-site well, MW-56 (**Table 5**).

VC Within biowall B2, VC was not detected. Downgradient of biowalls A1/A2 and B1/B2, VC was detected with a maximum detection of 120 µg/L at MWT-29, located approximately 34 feet downgradient of Biowall B2. The temporary production of VC is a product of reductive dechlorination of TCE. Thus, the elevated concentrations of VC downgradient of the biowalls and within biowalls B1 and C2 indicate that reductive dechlorination is occurring. VC was not detected at the off-site well, MW-56 (**Table 5**).

Other Chlorinated Ethenes:

1,2-Dichloroethane was detected above the Class GA groundwater standard of 0.6 µg/L at two locations; between biowalls B2 and C1 (2.6 µg/L) and at biowall C2 (1.3 µg/L) (**Table 4**). All other well locations were non-detect.

1,1-DCE was detected once (0.51 J µg/L). The detection was downgradient of the biowalls and upgradient of the ZVI wall. The detection was an estimated concentrations (J flag) and below its Class GA groundwater standard of 5 µg/L (**Table 4**).

T-1,2-DCE was detected at nine locations and exceeded its groundwater standard in two locations. The exceedances were in wells MWT-22 and PT-17, downgradient of all of the biowalls, at a concentration of 5.6 µg/L and 11 µg/L above its Class GA groundwater standard (5.0 µg/L). T-1,2-DCE was not detected in the off-site sentinel well (MW-56) (**Table 4**).

1,1-Dichloroethene (1,1-DCE) and trans-1,2-Dichloroethene (T-1,2-DCE) are degradation products of the reductive dehalogenation of TCE (**Figure 3**) (**Table 4**). During the breakdown of TCE, cis-1,2-DCE is the more prevalent intermediate followed by T-1,2-DCE and 1,1-DCE.

Other Compounds

Chloroform was detected in one well (PT-18A). Chloroform is not a historic COC; therefore, its detection is not believed to be associated with historic site operations.

4. BIOWALL RECHARGE EVALUATION

Recharge Evaluation Process

A recharge evaluation, described below, is the determination of the need to recharge a biowall segment. The evaluation consists of the following:

- Determining the need to recharge a biowall segment requires a review of chemical concentrations and geochemical parameters by an experienced professional. A specific, absolute set of conditions or parameter values are not appropriate to determine the need to recharge. Rather, a lines-of-evidence approach correlating a decrease in the efficiency of the system to degrade chloroethenes to geochemical evidence that indicates the cause is due to substrate depletion will be used. No single criterion is to be used to determine the efficacy of the biowall in the decision of whether recharge is required.
- The following parameters are evaluated annually using at least two consecutive rounds of sampling data to determine if recharge of the biowalls is necessary:
  - COC concentrations in the biowalls (e.g., MWT-27, MWT-28, and MWT-23). Detected COC concentrations that have increased above Class GA standards in consecutive rounds indicate that recharge may need to be considered. Concentrations within the biowalls, not at downgradient locations, will be used to make this evaluation so that the effectiveness of the wall itself is being measured without the interference of effects such as desorption and mixing.
  - Geochemical parameters, specifically ORP, TOC, and DO, in the biowalls (e.g., at MWT-27, MWT-28, and MWT-23). Benchmark values will be used initially to evaluate anaerobic conditions in the groundwater. The benchmarks are:
    - ORP < 50mV; preferred ORP < -100 mV
    - TOC > 20 mg/L
    - DO < 1.0 mg/L

The parameters described above are intended to be used as guidelines and will be considered in evaluating if, and when, a depletion of bioavailable organic substrate results in a rebound in geochemical redox conditions under which effective anaerobic degradation of chlorinated ethenes does not occur.

Recharge Evaluation for Round 21

Geochemical data presented in **Section 3** suggest that the values of geochemical parameters measured in Round 21 support the interpretation that reductive dechlorination is occurring in Biowalls B1/B2 and C1/C2 (**Exhibit 4.1**). The tables below show that the geochemical parameters for the wells within the biowalls meet, or are similar to, the benchmark values and that groundwater conditions remain reducing.

DO levels are less than (i.e., better than) the benchmark value of 1 mg/L at all wells indicating an environment adequate for anaerobic biodegradation (**Exhibit 4.1**). TOC levels are above (i.e., better than) the benchmark value of 20 mg/L at biowall B1 (MWT-27) indicating an adequate carbon and energy source

to drive dechlorination. In the last two rounds, the TOC concentration in biowall B2 has remained at a value approximately equal to, but below, the benchmark value. The TOC concentration in biowall C2 remains below the benchmark value. ORP levels remain above the preferred benchmark of -100 mV within the biowalls, but continue to be less than 50 mV. However, as discussed above, there is other evidence for methanogenesis (e.g., elevated methane levels in the biowalls); therefore, the fact that ORP values are not as low as previous sampling rounds does not appear to be impacting the overall effectiveness of the biowall system. The chlorinated ethene data in the biowalls indicate that ethene/ethane concentrations are typically higher within, and downgradient, of the biowalls. This indicates that reducing conditions are being maintained, thus breaking down TCE and forming its associated reduction products.

In Round 21, TCE was not detected in any of the wells in the biowalls (**Exhibit 4.2**). Concentrations of cDCE in the biowalls were well below the Class GA groundwater standard of 5 µg/L. Further, the ability of the biowalls to sustain reductive dechlorination is well established. During R21, the concentration of VC in biowall B1 exceeded its Class GA groundwater standard. This was the only COC that exceeded Class GA groundwater standards and has not occurred in multiple rounds.

Overall, the multiple lines-of-evidence approach that evaluates geochemical parameters together with the chemical analytical data indicates that conditions in the biowalls are sufficiently anaerobic to support reductive chlorination of chlorinated ethenes. Substrate in the biowalls has not been depleted and biodegradation continues to occur. At this time, although TOC levels are below the benchmark value at MWT-23 and slightly below at MWT-28, they remain high enough to support reductive chlorination. Low DO concentrations and negative ORPs indicate reducing conditions are being maintained with the current levels of TOC. Reductions in sulfate and the production of methane further indicate that highly anaerobic conditions are being sustained. There is no singular value that can be specified for any one parameter, in this case TOC, where crossing that value would indicate the need to recharge. Both an increasing trend in VOC concentrations and consistent negative trends in multiple geochemical parameters would need to be observed to consider that recharge is required.

However, some geochemical parameters were below benchmark values in the last couple of monitoring rounds. Additionally, some low variations in VOC concentrations were measured. Though recharge is not needed immediately based on the evaluation above, given the changes in the lines of evidence, a recharge event is being planned for 2017. The strategy for the recharge will be detailed in a work plan to be provided to the EPA and NYSEC for review.

Exhibit 4.1 – Geochemical Results from the Biowalls

Parameter	Benchmark Value	MWT-27 (Biowall B1)																			
		Q1	Q2	Q3	Q4	R5	R6	R7	R8	R9	R10	R11	R12	R13	R14	R15	R16	R17	R18	R19	R20	R21
ORP (mV)	< -100	-158	-145	-141	-166	-133	-126	-128	-102	-121	-111	-109	-71	-82	-120	-33	-66	-77	-105	-85	-77	-79
TOC (mg/L)	> 20	2050	1350	755	167	89	54	81.7	50	61	32	42	35	28	35	41	37	39	38	37	28	26.5
DO (mg/L)	< 1.0	0.25	0.08	0	0.06	0.18	0.13	0.06	0.15	0.05	0.05	0.01	0.08	0.03	0.03	0.04	0.22	0.52	0.08	0.14	0.29	0.13

Parameter	Benchmark Value	MWT-28 (Biowall B2)																			
		Q1	Q2	Q3	Q4	R5	R6	R7	R8	R9	R10	R11	R12	R13	R14	R15	R16	R17	R18	R19	R20	R21
ORP (mV)	< -100	-150	-113	-131	-151	-91	-95	-135	-148	-104	-100	-135	-126	-76	-73	-41	-49	-87	-88	-74	-18	-70
TOC (mg/L)	> 20	1775	171	309	92	49	28	28.2	25.5	21	12	17	12	18	25	25	24	19	18	24	19	16
DO (mg/L)	< 1.0	0.16	0.09	0	0.08	0.15	0.10	0.18	0.29	0.06	0.07	0.28	0.02	0.06	0.07	0.04	0.21	0.71	0.02	0.12	0.41	0.48

Parameter	Benchmark Value	MWT-23 (Biowall C2)																			
		Q1	Q2	Q3	Q4	R5	R6	R7	R8	R9	R10	R11	R12	R13	R14	R15	R16	R17	R18	R19	R20	R21
ORP (mV)	< -100	-122	-109	-87	-144	-129	-104	-117	-90	-115	-103	-136	-104	-71	-91	-102	-16	-56	-77	-80	-85	-64
TOC (mg/L)	> 20	260	210	303	151	29	20	15.6	17.4	11	5.9	1.5	6.3	4.8	11	4.1	5.5	4.7	5.6	3.4	4.8	4.7
DO (mg/L)	< 1.0	0.26	0.35	0	0.12	0.15	0.20	0.07	0.63	0.04	0.29	0.85	0.08	0.08	0.11	0.18	0.24	0.18	0.07	0.24	0.08	0.13

Exhibit 4.2 – VOC Concentrations in the Biowall Wells

	MWT-27 (Biowall B1)			MWT-28 (Biowall B2)			MWT-23 (Biowall C2)		
	TCE (µg/L)	**Cis-DCE (µg/L)**	**VC (µg/L)**	**TCE (µg/L)**	**Cis-DCE (µg/L)**	**VC (µg/L)**	**TCE (µg/L)**	**Cis-DCE (µg/L)**	**VC (µg/L)**
Q1	ND	ND	ND	ND	ND	ND	ND	**60**	**23**
Q2	ND	ND	ND	ND	ND	ND	ND	**11**	**4.8**
Q3	ND	ND	ND	ND	ND	ND	ND	3.1	ND
Q4	ND	ND	ND	ND	ND	ND	ND	3.6 J	**3.65**
R5	ND	ND	ND	ND	ND	ND	ND	ND	ND
R6	ND	ND	ND	ND	ND	ND	0.4	2.4	**2.8**
R7	ND	ND	ND	ND	ND	ND	ND	0.42 J	ND
R8	ND	ND	**3.1** J	ND	ND	ND	ND	0.47 J	ND
R9	ND	0.18 J	ND	ND	ND	ND	ND	0.41 J	ND
R10	0.51 J	1.1	**2.1**	ND	0.51 J	0.64 J	0.29 J	4.6	**5.3**
R11	ND	0.21 J	ND	ND	ND	ND	ND	0.57 J	0.33 J
R12	ND	1.4	**3.0**	ND	0.28 J	0.56 J	0.18 J	2.0	1.8
R13	ND	0.42 J	0.61 J	ND	ND	ND	ND	0.55 J	0.33 J
R14	ND	ND	ND	ND	ND	0.31 J	ND	1.9	1.65
R15	ND	ND	ND	ND	ND	ND	ND	3.3	**2.9**
R16	ND	0.48 J	0.84 J	ND	0.37 J	ND	ND	2.6	**2.5**
R17	ND	0.83 J	1.0	ND	ND	ND	ND	0.45 J	0.37 J
R18	ND	0.70 J	1.2	ND	0.19 J	ND	0.19 J	2.7	ND
R19	ND	0.67 J	ND	ND	ND	ND	ND	1.0	ND
R20	ND	0.87 J	ND	ND	ND	ND	ND	3.8	**3.4**
R21	ND	2.4	**3.1**	ND	ND	ND	ND	1.2	1.9

Notes:

1. ND = Not detected at the reporting limit.
2. NYSDEC Class GA Groundwater Standards: TCE = 5 µg/L; cis-DCE = 5 µg/L; VC = 2 µg/L.
3. Bolded values indicate exceedance above NYSDEC Class GA Groundwater Standards.

5. CONCLUSIONS

Based on the 21st round of LTM at the Ash Landfill OU since the installation of the full-scale biowalls, the Army makes the following conclusions:

- TCE, cDCE, and VC are present in the groundwater at concentrations above the Class GA groundwater standards;
- COCs do not exceed groundwater standards at the off-site sentinel well, MW-56;
- Chemical results indicate that the concentrations of chlorinated ethenes are decreasing as the groundwater plume passes through the biowall locations;
- Geochemical parameters indicate that groundwater redox conditions are conducive for reductive dechlorination to occur within the biowalls;
- Based on decreasing performance trends in some of the geochemical parameters, recharge of the biowall substrate will be considered; and
- The Army will continue to monitor the performance of the biowall system, including semi-annual periodic evaluations of the potential need to recharge the biowalls.

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Table 1
Groundwater Sample Collection
Round 21 - June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

	Monitoring Well Group			Laboratory Analysis				Field Test	
Monitoring Wells	**On-Site Plume Performance Monitoring**	**Biowall Process Monitoring**	**Off-Site Performance Monitoring**	VOC 8260B	TOC 9060A	MEE RSK-175	Sulfate EPA 300.1	Ferrous Iron (mg/L)	Manganese (mg/L)
PT-18A	X			X					
MWT-25	X			X					
MWT-26		X		X	X	X	X	X	X
MWT-27		X		X	X	X	X	X	X
MWT-28	X	X		X	X	X	X	X	X
MWT-29	X	X		X	X	X	X	X	X
MWT-22	X			X					
PT-22	X			X					
MWT-23	X	X		X	X	X	X	X	X
MWT-24	X			X					
PT-17	X			X	X	X	X	X	X
MWT-7	X			X	X	X	X	X	X
PT-24	X			X					
MW-56			X	X					

Notes:
1. All samples were analyzed for field parameters including pH, ORP, dissolved oxygen, conductivity, temperature and turbidity.
2. All samples were collected in Round 21 between June 14, 2016 and June 17, 2016.

Table 2
Groundwater Elevation Data
Round 21 - June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Monitoring Well	Top of Riser Elevation (ft)	Well Depth (rel. TOC) (ft)	LTM R21 - June 2016				Historical Data		
			Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Groundwater Elevation (ft)		
							Maximum	Minimum	Range
PT-18A	659.05	12.85	6/14/2016	3.66	9.19	649.86	653.25	649.65	3.60
MWT-25	654.51	13.25	6/14/2016	5.70	7.55	646.96	650.65	645.93	4.72
MWT-26	652.19	13.22	6/14/2016	6.15	7.07	645.12	648.92	644.58	4.34
MWT-27	652.99	12.90	6/14/2016	5.32	7.58	645.41	648.60	644.27	4.33
MWT-28	652.69	12.85	6/14/2016	4.95	7.90	644.79	648.31	644.20	4.11
MWT-29	651.82	13.10	6/14/2016	4.94	8.16	643.66	647.83	643.18	4.65
MWT-22	650.66	14.90	6/14/2016	7.16	7.74	642.92	648.13	642.29	5.84
PT-22	648.61	11.81	6/14/2016	2.96	8.85	639.76	644.30	637.47	6.83
MWT-23	646.77	13.70	6/14/2016	4.62	9.08	637.69	640.61	636.40	4.21
MWT-24	641.56	13.00	6/14/2016	5.38	7.62	633.94	635.84	632.11	3.73
PT-17	640.14	11.65	6/14/2016	6.01	5.64	634.50	637.50	632.74	4.76
MWT-7	638.34	13.64	6/14/2016	7.60	6.04	632.30	633.58	626.58	7.00
PT-24	636.40	11.88	6/14/2016	6.61	5.27	631.13	632.76	627.80	4.96
MW-56	630.51	6.88	6/14/2016	2.33	4.55	625.96	627.58	624.39	3.19

Table 3
Groundwater Geochemical Data
Years 1 - 10
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Well ID	Location Description	Sample ID	Sample Round	pH	Turbidity (NTU)	Specific Conductance (mS/cm)	DO (mg/L)	ORP (mV)	TOC (mg/L)	Sulfate (mg/L)	Ethane (ug/L)	Ethene (ug/L)	Methane (ug/L)	Manganese (ug/L)	Ferrous Iron (ug/L)
PT-18A	Upgradient of walls	ALBW20059	1Q2007	6.63	141	1.69	1.33	93							
		ALBW20074	2Q2007	6.44	110	2.87	0.76	-177							
		ALBW20088	3Q2007	6.71	5	1.66	0	-23							
		ALBW20103	4Q2007	6.41	0.0	1.25	0.04	-5							
		ALBW20117	5R2008	6.36	1.9	1.75	0.22	-10						8.2	> 3.3
		ALBW20132	6R2008	6.58	0.56	2.04	1.76	83							
		ALBW20147	7R2009	6.77	0.45	2.01	0.12	66							
		ALBW20162	8R2009	6.71	0.00	2.04	0.62	154							
		ALBW02177	9R2010	6.7	1.00	2.05	0.1	62						1.5	0.15
		ALBW20192	10R2010	6.66	1.50	1.25	0.16	84							
		ALBW20207	11R2011	6.62	3.30	1.27	0.19	-17							
		ALBW20222	12R2011	6.62	0.40	1.75	0.09	59.9							
		ALBW20237	13R2012	6.80	4.25	1.06	0.12	78							
		ALBW20252	14R2012	6.84	1.83	1.33	4.22	74							
		ALBW20265	15R2013	6.83	2.49	1.46	0.13	199							
		ALBW20280	16R2013	6.76	0.92	1.11	1.18	201							
		ALBW20296	17R2014	6.95	0.77	1.20	0.15	76							
		ALBW20312	18R2014	7.07	0.93	1.23	2.99	107							
		ALBW20328	19R2015	6.66	1.10	1.40	0.67	121							
		ALBW20344	20R2015	7.17	0.74	1.12	0.86	114							
		ALBW20360	21R2016	7.04	1.39	1.40	0.25	112							
MWT-25	Upgradient of Biowall A	ALBW20064	1Q2007	8	9.6	0.290	2.83	63							
		ALBW20079	2Q2007	7.27	14	2.20	2.8	52							
		ALBW20093	3Q2007	7.36	6.2	2.43	4.14	100							
		ALBW20108	4Q2007	6.9	0	1.20	0.21	65							
		ALBW20123	5R2008	6.91	0.52	1.47	0.15	-41						1.4	0.75
		ALBW20138	6R2008	6.69	1.32	1.36	2.91	90							
		ALBW20153	7R2009	7.03	1.6	1.46	0.1	-31							
		ALBW20168	8R2009	7.21	0	0.792	3.35	98							
		ALBW20183	9R2010	7.06	0.7	1.48	0.2	-116							
		ALBW20198	10R2010	7.11	2.59	1.23	0.48	-94							
		ALBW20213	11R2011	6.72	13	1.13	0.03	13							
		ALBW20228	12R2011	7	5.2	1.50	1.5	-54.9							
		ALBW20243	13R2012	7.13	1.71	0.934	0.01	18							
		ALBW20258	14R2012	7.5	3.08	0.876	6.63	32							
		ALBW20271	15R2013	6.93	1.74	1.11	0.08	-5							
		ALBW20286	16R2013	7.49	2.44	1.00	2.77	185							
		ALBW20302	17R2014	7.36	0.92	1.46	0.09	-19							
		ALBW20318	18R2014	7.57	1.71	1.22	4.7	87							
		ALBW20334	19R2015	7.05	0.52	1.25	0.27	84							
		ALBW20350	20R2015	7.35	4.75	1.84	0.49	112							
		ALBW20366	21R2016	6.92	1.04	1.72	0.41	23							
MWT-26	**Upgradient of Biowalls B1/B2**	ALBW20066	1Q2007	6.89	10	2.01	1.84	-3	3.9 J	958	ND	ND	ND		
		ALBW20081	2Q2007	7.26	9	1.90	0.48	-135	15.2	738	0.4	7.8	210	2.1	> 3.3
		ALBW20095	3Q2007	6.89	2.2	1.94	0.21	-170	10.3	473	1	13	390	3.1	> 3.3
		ALBW20111	4Q2007	7.08	50	1.90	0.89	-40	6.1	1060	0.16	0.4	44	0.0	1.09
		ALBW20126	5R2008	7.05	0.67	1.88	0.31	-71	5.6	600	0.82	2.9	210	1.3	0.81
		ALBW20141	6R2008	7.01	28.7	1.58	3.54	60	4.4	541	0.046	0.028	10	0.6	0.22
		ALBW20156	7R2009	6.95	2.7	1.75	0.34	-11	6.9	570	3.2	2.7	1,100	0.5	0.71
		ALBW20171	8R2009	7.01	10	2.45	4.66	71	5.6	912	2.2	1.8	610	0.7	0.18
		ALBW20186	9R2010	6.99	1.4	2.04	0.14	-81	4.6	680	2.2	0.71	740	1.7	2.67
		ALBW20202	10R2010	6.77	0.6	1.71	0.5	109	5.5	690	3.7	3.3	1600	0.0	0.13
		ALBW20216	11R2011	6.64	2.4	1.67	0.07	-31	6.3	510	4.5	1	960	6.3	0.89
		ALBW20232	12R2011	7.05	2.2	1.87	0.54	12.1	4.5	860	0.23	ND	39	0.5	0.0
		ALBW20246	13R2012	7.26	8.72	1.02	0.48	2	4.4	640	1	0.5	230	0.6	0.09
		ALBW20262	14R2012	7.37	1.5	0.909	5.47	103	4.5	430	0.096	0.069	9.4	0.0	0.01
		ALBW20274	15R2013	6.91	1.24	0.834	0.13	24	4.1	250	0.69	0.54	130	1.0	0.02
		ALBW20289	16R2013	7.42	3.89	1.40	2.88	140	5.4	610	0.52	0.2	61	0.3	0.00
		ALBW20306	17R2014	6.80	17.5	1.72	0.36	61	4.7	680	0.92	0.15 J	240	1.2	0.04
		ALBW20321	18R2014	7.31	1.47	1.16	5.5	154	4.4	460	0.62	0.24	60	0.0	0.0
		ALBW20337	19R2015	6.83	2.22	1.88	0.27	143	5.5	680	0.43	0.13 J	83	0.7	0.01
		ALBW20354	20R2015	7.25	2.26	1.77	1.13	59	5.9	640	1.1	0.27	140	0.2	0.0
		ALBW20369	21R2016	7.32	0.86	1.5	0.23	77	5.0	590	0.69	0.18 J	170	0.8	0.02
MWT-27	**In Biowall B1**	ALBW20067	1Q2007	6.34	120	5.31	0.25	-158	2,050 J	ND	ND	ND			
		ALBW20082	2Q2007	6.65	87	4.37	0.08	-145	1350	ND	0.15	2.7	15,000	> 22	> 3.3
		ALBW20096	3Q2007	6.59	154	3.35	0	-141	755	1.9 J	0.081	0.33	13,500	> 22	> 3.3
		ALBW20112	4Q2007	6.43	58	5.76	0.06	-166	167	31.7	ND	0.014 J	13,000	> 22	2.19
		ALBW20127	5R2008	6.49	40	3.07	0.18	-133	88.9	ND	2.3	0.049	13,000	> 22	3.23
		ALBW20142	6R2008	5.95	24.5	2.59	0.13	-126	53.5	24	1.6	0.13	15,000	> 22	3.05
		ALBW20157	7R2009	6.68	38	2.99	0.06	-128	81.7	0.93 J	5.1	0.15	14,000	22	1.88
		ALBW20172/73	8R2009	6.32	5.1	2.38	0.15	-102	50.0	14.0	4.35	1.2	15,500	9	1.26
		ALBW20187	9R2010	6.52	1.4	2.55	0.05	-121	61	0.95 J	3.8	0.12	13,000	> 22	2.54
		ALBW20203	10R2010	6.42	8.91	2.22	0.05	-111	32	25.0	3	0.88	18,000	48	3.30
		ALBW20217/18	11R2011	6.3	3.2	1.75	0.01	-109	42	0.69 J	6.2	0.077	14,000	>22	>3.3
		ALBW20233	12R2011	5.55	16	1.98	0.08	-71	35	19.0	2	1.6	16,000	>22	1.23
		ALBW20247	13R2012	6.68	15.3	1.81	0.03	-82	28	1.4	8.4	0.68	14,000	22	1.80
		ALBW20263	14R2012	6.62	15.6	2.26	0.03	-120	35	2.5	0.88	0.051	13,000	47.5	2.17
		ALBW20275/76	15R2013	6.59	9.33	2.14	0.04	-33	41	4.1	2.15	ND	12,500	>47.5	2.63
		ALBW20290	16R2013	6.43	15.0	1.85	0.22	-66	37 J	5.2	1.4	0.16 J	20,000	19.2	2.75
		ALBW20307	17R2014	6.53	18.5	2.09	0.52	-77	39	6.6	5.3	0.79	16,000	>22	>3.3
		ALBW20322/23	18R2014	6.79	8.29	1.77	0.08	-105	38	36.5	2.2	0.335	12,000	>22	>3.3
		ALBW20338	19R2015	6.42	14.8	1.53	0.14	-85	37	5.7	4	0.56	16,000	>22	0.74
		ALBW20355	20R2015	6.60	12.2	1.73	0.29	-77	28	7.9	1.4	0.18 J	16,000	>47.5	3.14
		ALBW20370/371	21R2016	6.62	9.26	1.73	0.13	-79	26.5	11	4.5	0.965	17,000	>47.5	3.29
MWT-28	**In Biowall B2**	ALBW20068	1Q2007	7.5	163	0.610	0.16	-150	1,775 J	1.7	ND	ND	12,500 J		
		ALBW20083	2Q2007	6.6	21	2.30	0.09	-113	171	ND	0.67	0.48	19,000	7.5	> 3.3
		ALBW20098	3Q2007	6.56	100	2.74	0	-131	309	ND	0.01 J	0.057	11,000	> 22	> 3.3
		ALBW20113	4Q2007	6.48	10	1.72	0.08	-151	92	ND	0.014 J	ND	11,000	> 22	2.15
		ALBW20128	5R2008	6.31	14	2.16	0.15	-91	49.2	ND	0.65	0.044	12,000	> 22	> 3.3
		ALBW20144	6R2008	5.76	17	1.58	0.10	-95	27.9	48.3	2	0.12	19,000	5.3	1.98
		ALBW20158/59	7R2009	6.49	8.5	1.73	0.18	-135	28.2	ND	1.8	0.064	13,000	20.8	2.87
		ALBW20174	8R2009	6.4	10.8	1.88	0.29	-148	25.5	3.16	1.6	0.12	15,000	6.5	2.15
		ALBW20188/89	9R2010	6.36	5.5	1.62	0.06	-104	21	ND	1.55	0.059	13,500	18.6	0.57
		ALBW20204	10R2010	6.28	4.5	0.802	0.07	-100	12	4.8	1.4	0.17	12,000	5.8	2.58
		ALBW20219	11R2011	6.14	3.93	1.44	0.28	-135	17	0.63 J	0.9	0.0085 J	8,800	8.9	>3.3
		ALBW20234	12R2011	5.76	6.2	0.773	0.02	-125.9	12	19	1.6	ND	12,000	1.1	0.48
		ALBW20248/49	13R2012	6.46	7.41	1.16	0.06	-76	18	0.58	3.1	0.069	14,000	16.4	1.65
		ALBW20264	14R2012	6.27	16.3	1.38	0.07	-73	25	1.1	0.38	0.074	11,000	36	2.79
		ALBW20277	15R2013	6.27	6.33	1.33	0.04	-41	25	1.1 J	1.6	ND	14,000	>47.5	>3.3
		ALBW20291/92	16R2013	6.37	7.01	1.42	0.21	-49	24 J	2.5 U	1.45	0.24 J	19,500	4.3	2.74
		ALBW20308	17R2014	6.28	11.5	1.22	0.71	-87	19	1.3 U	2.8	0.0068 J	15,000	17.5	>3.3
		ALBW20324	18R2014	6.55	6.84	1.44	0.02	-88	18	11	0.35	0.049 J	12,000	7.4	2.13
		ALBW20339/40	19R2015	6.35	4.86	1.29	0.12	-74	24	0.84 J	1.55	ND	14,500	36.2	>3.3
		ALBW20356	20R2015	6.71	9.27	1.41	0.41	-18	19	ND	0.29	ND	14,000	34.3	3.13
		ALBW20372	21R2016	6.35	3.32	1.21	0.48	-70	16	0.77 J	1.7	ND	15,000	42.6	>3.3

Table 3
Groundwater Geochemical Data
Years 1 - 10
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Well ID	Location Description	Sample ID	Sample Round	pH	Turbidity (NTU)	Specific Conductance (mS/cm)	DO (mg/L)	ORP (mV)	TOC (mg/L)	Sulfate (mg/L)	Ethane (ug/L)	Ethene (ug/L)	Methane (ug/L)	Manganese (ug/L)	Ferrous Iron (ug/L)
MWT-29	Downgradient of Biowall B2	ALBW20070	1Q2007	6.49	7.2	2.10	0.33	-76	25.1 J	113	ND	ND	ND		
		ALBW20084/5	2Q2007	6.8	1.7	2.21	0.39	-53	36.7	173	25	150	8,100	7.5	> 3.3
		ALBW20099	3Q2007	6.64	1.8	1.68	0.11	-79	15.7	151	13	160	2,800	8.1	2.84
		ALBW20114	4Q2007	7.04	12.2	1.88	0.21	-101	20.9	289	19	200	2,600	8.6	> 3.3
		ALBW20129/30	5R2008	6.44	2.7	1.85	0.17	-115	14.1	174	14.5	140	3,100	0.0	> 3.3
		ALBW20145	6R2008	6.57	3.69	1.58	1.32	67	13.6	312	14	19	2,700	3.3	0.20
		ALBW20160	7R2009	6.8	1.9	1.80	0.15	-105	11.8	300	10	47	3,000	6.8	2.97
		ALBW20175	8R2009	6.87	0	2.05	0.58	-75	8.2	644	6.7	12	1,500	6.3	0.96
		ALBW20190	9R2010	6.77	2	1.74	0.06	-86	10	170	18	88	5,400	9.1	2.54
		ALBW20205	10R2010	6.71	1.07	1.31	0.56	22	7.4	300	5.1	7.9	3,100	6.4	2.60
		ALBW20220	11R2011	6.55	2.8	1.37	0.05	-90	7.7	170	8.3	47	3,100	12.1	0.03
		ALBW20235	12R2011	6.26	1.9	0.898	0.29	-30.2	4.9	210	1.7	7.3	760	1	0.04
		ALBW20250	13R2012	6.78	1.05	1.05	0.07	-29	8.2	95	10	38	5,200	19.6	2.88
		ALBW20265	14R2012	6.96	1.58	0.606	4.99	312	4.8	130	0.58	0.8	180	1.2	0.00
		ALBW20278	15R2013	6.65	1.72	0.960	0.07	25	5.8	84	2.9	6.6	2,500	0.09	0.93
		ALBW20293	16R2013	6.91	4.46	0.844	3.43	54	6.2	130	1.5	3.8	1,700	2.0	0.05
		ALBW20309	17R2014	6.59	0.96	1.60	0.13	-28	5.7	97	9.1	45	6,100	7.9	>3.3
		ALBW20325	18R2014	7.15	1.67	0.712	7.55	81	4.7	120	0.34	0.87	160	0.7	0.11
		ALBW20341	19R2015	6.62	0.81	1.37	0.15	-30	8.6	84	8.3	27	5,400	8.0	>3.3
		ALBW20357	20R2015	6.89	1.55	0.96	0.83	-16	6.1	180	2.3	7.4	1,600	3.1	0.54
		ALBW0373	21R2016	6.65	0.71	1.54	0.28	-60	6.5	90	5.6	17	6,200	18.4	>3.3
MWT-22	Downgradient of Biowall B2	ALBW20071	1Q2007	7.7	4.5	0.130	0.09	-80							
		ALBW20075	2Q2007	6.72	41	2.16	0.3	-65							
		ALBW20100	3Q2007	6.45	2.7	2.03	0.05	-107							
		ALBW20115	4Q2007	6.53	7.5	1.81	0.18	-132							
		ALBW20121	5R2008	6.38	14	2.21	0.3	-34						18.2	> 3.3
		ALBW20136	6R2008	6.44	8.17	1.86	0.57	-19							
		ALBW20151	7R2009	6.59	13	2.14	0.31	-91							
		ALBW20166	8R2009	6.5	15	0.898	0.34	-65							
		ALBW20181	9R2010	6.52	16.8	2.20	0.22	-63							
		ALBW20196	10R2010	6.39	6.8	1.34	0.07	-58							
		ALBW20211	11R2011	6.2	3.6	1.27	0.16	-71							
		ALBW20226	12R2011	5.65	7.7	1.81	0.05	10							
		ALBW20241	13R2012	6.41	8.78	1.37	0.17	-27							
		ALBW20256	14R2012	6.54	12	1.53	0.11	-55							
		ALBW20269	15R2013	6.48	4.63	1.32	0.47	-11							
		ALBW20281	16R2013	6.47	20.4	1.55	0.15	-21							
		ALBW20300	17R2014	6.58	8.99	1.35	0.32	-33							
		ALBW20316	18R2014	7.33	10.7	1.36	0.17	-46							
		ALBW20332	19R2015	6.78	3.46	1.52	0.45	-14							
		ALBW20348	20R2015	6.55	4.25	1.34	1.05	-77							
		ALBW20364	21R2016	6.31	4.93	1.47	0.24	-37							
PT-22	Between Biowalls B and C	ALBW20060	1Q2007	7.70	4.5	0.130	0.09	-80							
		ALBW20086	2Q2007	6.78	7	1.18	0.78	-54							
		ALBW20089	3Q2007	6.67	0	1.44	0.09	-97							
		ALBW20104	4Q2007	6.73	5.1	1.26	0.17	-166							
		ALBW20118	5R2008	6.69	7.4	1.38	0.29	-119						0.3	1.38
		ALBW20133	6R2008	6.79	1.96	1.20	0.69	-37							
		ALBW20148	7R2009	6.76	11	1.53		-123							
		ALBW20163	8R2009	6.74	6.3	1.45	1.0	-73							
		ALBW20178	9R2010	6.87	3.6	1.39	0.4	-75							
		ALBW20193	10R2010	6.75	0.8	1.14	0.18	15							
		ALBW20208	11R2011	6.65	2	0.877	0.39	-62							
		ALBW20223	12R2011	5.95	0.38	1.57	0.27	205.8							
		ALBW20238	13R2012	6.74	8.4	0.904	2.50	12							
		ALBW20253	14R2012	6.98	1.44	0.731	5.36	201							
		ALBW20266	15R2013	6.77	0.82	0.927	0.08	82							
		ALBW20284	16R2013	6.62	0.38	0.942	1.54	203							
		ALBW20297	17R2014	6.74	2.42	1.05	1.48	61							
		ALBW20313	18R2014	7.07	2.83	0.921	6.66	134							
		ALBW20329	19R2015	7.02	0.59	1.09	0.11	121							
		ALBW20345	20R2015	7	1.29	0.981	1.59	131							
		ALBW20361	21R2016	6.71	0.88	1.21	0.64	107							
MWT-23	In Biowall C2	ALBW20065	1Q2007	7.2	5	0.200	0.26	-122	260 J	ND	ND	ND	12,000		
		ALBW20080	2Q2007	6.51	30	1.80	0.35	-109	210	ND	45	5.9	23,000	5.4	2.73
		ALBW20094	3Q2007	6.3	69.3	1.82	0	-87	303	ND	4.1	0.28	18,000	> 22	2.99
		ALBW20109	4Q2007	6.32	21	2.21	0.12	-144	151	2.8	0.58	0.35	16,000	> 22	2.32
		ALBW20125	5R2008	6.27	29	1.54	0.15	-129	28.4	ND	0.53	0.048	18,000	> 22	> 3.3
		ALBW20140	6R2008	6.44	32	1.86	0.20	-104	20.1	6.3	4.6	1.2	19,000	> 22	2.75
		ALBW20155	7R2009	7.72	16	1.50	0.07	-117	15.6	ND	1.6	0.16	21,000	22	2.08
		ALBW20170	8R2009	6.78	10	2.10	0.63	-90	17.4	ND	1	0.058	18,000	7	3.3
		ALBW20185	9R2010	6.38	9	1.57	0.04	-115	11	ND	2.4	0.038	18,000	>22	1.71
		ALBW20200/201	10R2010	6.41	2.8	1.07	0.29	-103	5.9	16	16	2.85	16,000	13	> 3.3
		ALBW20215	11R2011	6.21	5.97	1.20	0.85	-136	6.2	1.5	2.3	0.1	15,000	8	>3.3
		ALBW20230/231	12R2011	5.64	6.7	1.00	0.08	-104.1	6.3	14	8.9	1.2	16,000	12.6	1.17
		ALBW20245	13R2012	6.52	6.14	0.915	0.08	-71	4.8	1.5	5	0.26	18,000	31.1	3.3
		ALBW20260	14R2012	6.33	8.56	1.07	0.11	-91	11	13	2.5	0.63	15,500	46.1	2.18
		ALBW20273	15R2013	6.60	6.81	0.929	0.18	-102	4.1	15	7.0	2.6	14,000	8.7	>3.3
		ALBW20288	16R2013	6.59	5.23	1.05	0.24	-16	5.5	10	7.0	1.7	15,000	15.3	>3.3
		ALBW20304/05	17R2014	6.40	3.17	1.25	0.18	-56	4.7	1.4 J	11 J	0.13 J	16,500	4.0	>3.3
		ALBW20320	18R2014	6.69	7.93	1.21	0.07	-77	5.6	98	5.4	2.2	13,000	4.8	3.21
		ALBW20336	19R2015	6.46	4.08	0.989	0.24	-80	3.4	13	3.1	2	14,000	5.1	>3.3
		ALBW20352	20R2015	6.86	4.48	0.929	0.08	-85	4.8	9.5	7.7	2.4	13,000	4.9	>3.3
		ALBW20368	21R2016	6.90	4.31	0.960	0.13	-64	4.7	5.9	5.4	0.82	14,000	3.5	2.61
MWT-24	Downgradient of Biowalls C1/C2	ALBW20063	1Q2007	7.02	10	0.762	0.27	-160							
		ALBW20078	2Q2007	6.91	59	1.08	0.32	-146							
		ALBW20092	3Q2007	6.8	5.4	1.48	0.03	-115							
		ALBW20107	4Q2007	6.81	134	1.32	0.41	-114							
		ALBW20122	5R2008	6.65	45	1.21	0.35	-43						9.1	1.54
		ALBW20137	6R2008	6.40	10	1.31	0.09	40							
		ALBW20152	7R2009	6.81	6.7	1.34	0.11	-20							
		ALBW20164	8R2009	6.61	23	0.558	1.31	59							
		ALBW20182	9R2010	6.63	6.8	1.45	0.06	-21							
		ALBW20197	10R2010	6.78	8.9	0.919	0.14	10							
		ALBW20212	11R2011	6.67	75	0.735	0.39	27							
		ALBW20227	12R2011	6.56	8.67	0.627	0.10	46.2							
		ALBW20242	13R2012	7.22	10.2	0.747	0.11	3							
		ALBW20257	14R2012	6.69	9.47	0.689	0.55	181							
		ALBW20270	15R2013	6.78	4.44	0.765	0.05	42							
		ALBW20285	16R2013	6.97	19.7	0.801	0.12	38							
		ALBW20301	17R2014	7.07	8.88	1.21	0.11	15							
		ALBW20317	18R2014	7.57	9.16	0.826	1.03	95							
		ALBW20333	19R2015	7.08	9.35	0.982	0.06	51							
		ALBW20349	20R2015	6.99	9.79	0.901	0.13	55							
		ALBW20365	21R2016	6.43	9.80	0.970	0.09	44							

Table 3
Groundwater Geochemical Data
Years 1 - 10
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Well ID	Location Description	Sample ID	Sample Round	pH	Turbidity (NTU)	Specific Conductance (mS/cm)	DO (mg/L)	ORP (mV)	TOC (mg/L)	Sulfate (mg/L)	Ethane (ug/L)	Ethene (ug/L)	Methane (ug/L)	Manganese (ug/L)	Ferrous Iron (ug/L)
PT-17[1]	Downgradient of biowalls	ALBW20058	1Q2007	8	3.8	92.0	0.23	-111							
		ALBW20073	2Q2007	7.1	14	0.729	0.76	-151							
		ALBW20087	3Q2007	6.99	0.4	0.732	0.9	-157							
		ALBW20102	4Q2007	7.12	8.7	2.00	NS	-24							
		ALBW20116	5R2008		70		0.24		6	15.2	98	66	5700		
		ALBW20131	6R2008	6.68	0.85	0.796	0.30	26	2.6	45.8	6.9	6.6	380	2.8	0.43
		ALBW20146	7R2009	7.19	0.2	1.00	0.30	-20	4.9	28	50	56	8300	7.5	0.53
		ALBW20161	8R2009	6.75	4	0.345	0.58	-52	2.4	46.2	9.9	5	1,500	2.1	0.07
		ALBW20176	9R2010	6.73	0.9	0.816	0.11	-13	2.4	36	16	20	4,300	5.8	0.29
		ALBW20191	10R2010	6.72	0.45	0.619	0.21	42	1.5	31	4.8	3.5	900	4.0	0.06
		ALBW20206	11R2011	6.57	4	0.573	0.85	-22	3.4	24	1.8	3.8	780	>22	0.64
		ALBW20221	12R2011	6.73	3.03	0.686	2.63	91	1.6	27	1.7	2.4	810	0.6	0.01
		ALBW20236	13R2012	7.09	2.8	0.688	0.17	28	2.8	25	10	12	8,200	4.6	0.0
		ALBW20251	14R2012	6.74	0.51	0.569	3.44	52	1.7	35	2.2	2.4	810	2.2	0.08
		ALBW20264	15R2013	6.94	1.36	0.679	0.24	74	1.2	27	1.1	0.69	780	3.4	0.0
		ALBW20279	16R2013	6.83	0.64	0.663	0.75	62	2.0	31	1.5	1.4	960	2.6	0.10
		ALBW20295	17R2014	6.18	0.55	0.908	0.07	35	2.9	20	4.5	6.5	5,700	4.5	0.14
		ALBW20311	18R2014	6.87	0.94	0.687	0.48	28	1.7	29	2.5	2.0	1,600	6.5	0.08
		ALBW20327	19R2015	7.09	0.46	0.780	0.22	40	1.6	25	1.6	1.8	1,600	5.9	0.05
		ALBW20343	20R2015	7.13	0.93	0.526	0.79	111	1.7	42	0.13 J	0.044 J	21	0.5	0
		ALBW20359	21R2016	6.77	0.79	0.830	0.06	12	2.9	18	1.9	1.5	3,400	3.5	0.18
MWT-7	Immediately upgradient of ZVI wall	ALBW20062	1Q2007	6.80	19.6	0.581	0.01	62							
		ALBW20077	2Q2007	6.95	8	0.763	0.76	52							
		ALBW20091	3Q2007	6.91	4	0.586	0.19	22							
		ABLW20106	4Q2007	6.88	0	0.900	0.16	14							
		ALBW20120	5R2008	6.85	15	0.974	0.43	37	2.3	29.1	6.7	2	400	0.2	0.09
		ALBW20135	6R2008	6.85	7.37	0.859	0.28	66	29.1	3	11	0.27	670	0.8	0.16
		ALBW20150	7R2009	7.61	2.6	0.786	0.05	16	3.1	27	7.8	0.76	1100	0	0.05
		ALBW20165	8R2009	7.12	0.9	0.555	0.46	32	4.5	29.3	17	0.52	2,900	0.01	0.14
		ALBW20180	9R2010	6.85	1.35	1.04	0.02	-21	1.5	29	9	0.55	1,700	0.2	0.19
		ALBW20195	10R2010	6.85	3.3	0.758	0.06	35	1.3	31	4.5	0.2	400	1.1	0.18
		ALBW20210	11R2011	6.70	0.85	0.784	0.08	-85	2	39	4.9	0.21	1,600	0.4	0.45
		ALBW20225	12R2011	6.56	3.9	0.619	0.17	197	1.7	26	0.84	ND	79	0.2	0.05
		ALBW20240	13R2012	6.86	3.67	0.638	0.24	-35	1.6	28	3.1	0.33	1,600	0.1	0.0
		ALBW20255	14R2012	6.85	1.74	0.597	2.84	34	1.6	29	0.64	0.067	96	0	0.1
		ALBW20268	15R2013	6.82	1.88	0.663	0.34	68	0.89 J	31	0.5	ND	160	0.0	0.0
		ALBW20283	16R2013	6.95	3.63	0.833	0.32	66	2.0	26	1.2	0.18 J	1,000	0.8	0.27
		ALBW20299	17R2014	6.26	3.16	0.946	0.55	63	1.4	23	1.2	0.19 J	510	0.6	0.0
		ALBW20315	18R2014	7.00	2.35	0.954	0.61	81	2.0	23	1.1	0.095 J	1300	0.7	0.0
		ALBW20331	19R2015	7.14	0.25	0.798	0.08	30	0.86 J	23	0.74 J	0.084 J	160	0.7	0.3
		ALBW20347	20R2015	7.09	1.12	0.719	0.23	57	1.8	22	0.84	0.13 J	440	0.2	0.09
		ALBW20363	21R2016	6.92	0.92	0.729	0.10	64	2	26	0.36	0.054 J	160	1.1	0.09
PT-24	Downgradient of ZVI wall	ALBW20061	1Q2007	8.10	10	70.00	0.37	-59							
		ALBW20076	2Q2007	7.58	0	0.464	2.2	-59							
		ALBW20090	3Q2007	7.22	1.3	0.557	0.13	-80							
		ALBW20105	4Q2007	7.35	9.7	2.38	0.19	-46							
		ALBW20119	5R2008	6.99	4.3	0.900	0.16	-104						0.5	0.55
		ALBW20134	6R2008	6.84	5.8	0.656	0.11	-10							
		ALBW20149	7R2009	7.14	4.1	0.679	0.05	-101							
		ALBW20164	8R2009	7.32	1	0.410	0.34	-192						1.9	0.2
		ALBW20179	9R2010	7.07	8.3	0.780	0.19	-37							
		ALBW20194	10R2010	7.05	6.14	0.568	0.09	-29							
		ALBW20208	11R2011	6.69	1.6	0.528	0.82	-16							
		ALBW20224	12R2011	6.79	0.48	0.391	0.13	26.2							
		ALBW20239	13R2012	7.47	8.9	0.554	0.14	-55							
		ALBW20254	14R2012	6.95	1.23	0.428	1.19	77							
		ALBW20267	15R2013	7.20	1.68	0.484	0.06	10							
		ALBW20282	16R2013	6.97	2.12	0.538	0.19	19							
		ALBW20298	17R2014	6.93	1.12	0.935	0.05	18							
		ALBW20314	18R2014	7.84	8.65	0.008	0.15	83							
		ALBW20330	19R2015	7.31	0.69	0.748	0.04	6							
		ALBW20346	20R2015	6.94	1.64	0.628	0.14	69							
		ALBW20362	21R2016	7.35	1.92	0.692	0.06	9							
MW-56 [2]	Off-site well	ALBW20072	1Q2007	6.85	3.30	0.462	0.37	-102							
		ALBW20101	3Q2007	6.90	0.00	0.603	NS	-65							
		ALBW20124	5R2008	6.73	2.00	0.763	0.18	-132						0.4	1.18
		ALBW20139	6R2008	6.85	6.00	0.545	0.81	-125							
		ALBW20154	7R2009	7.01	0.10	0.623	0.23	-186							
		ALBW20169	8R2009	6.59	7.30	0.311	1.86	-149							
		ALBW20184	9R2010	6.85	3.19	0.403	0.16	-131							
		ALBW20199	10R2010	6.88	1.26	0.659	0.32	-105							
		ALBW20214	11R2011	6.89	4.80	0.662	0.21	-105							
		ALBW20229	12R2011	7.15	5.50	0.415	0.45	-74.2							
		ALBW20244	13R2012	7.00	1.20	0.520	0.23	-283							
		ALBW20259	14R2012	6.95	3.16	0.504	0.93	-69							
		ALBW20272	15R2013	7.00	1.65	0.509	0.42	-192							
		ALBW20287	16R2013	6.86	1.49	0.534	0.38	-77							
		ALBW20303	17R2014	7.00	1.33	0.696	0.44	-99							
		ALBW20319	18R2014	7.11	3.03	0.687	0.52	-119							
		ALBW20335	19R2015	6.54	12.9	0.555	0.53	-119							
		ALBW20351	20R2015	6.89	3.04	0.588	0.55	-108							
		ALBW20367	21R2016	6.63	1.87	0.792	0.22	-87							

Notes:
> = The concentration exceeded the range of the Hach DR/850 Colorimeter field kit.
J = the reported value is an estimated concentration.
ND = Non-detect.
NS = Not sampled; water level was below the indicator probe.

1Q2007 - First round of LTM (January 2007)
2Q2007 - Second round of LTM (March 2007)
3Q2007 - Third round of LTM (June 2007)
4Q2007 - Fourth round of LTM (November 2007)
5R2008 - Fifth Round of LTM (June 2008)
6R2008 - Sixth Round of LTM (December 2008)
7R2009 - Seventh Round of LTM (June 2009)
8R2009 - Eighth Round of LTM (December 2009)
9R2010 - Ninth Round of LTM (June 2010)
10R2010 - Tenth Round of LTM (December 2010)
11R2011 - Eleventh Round of LTM (July 2011)
12R2011 - Twelfth Round of LTM (December 2011)
13R2012 - Thirteenth Round of LTM (June 2012)
14R2012 - Fourteenth Round of LTM (December 2012)
15R2013 - Fifteenth Round of LTM (July 2013)
16R2013 - Sixteenth Round of LTM (December 2013)
17R2014 - Seventeenth Round of LTM (June 2014)
18R2014 - Eighteenth Round of LTM (December 2014)
19R2015 - Nineteenth Round of LTM (June 2015)
20R2015 - Twentieth Round of LTM (December 2015)
21R2016 - Twenty-first Round of LTM (June 2016)

Empty cells indicate that the specified analysis was not completed for that well. The bolded wells are the five wells included in the biowall process monitoring group.
Analysis of TOC, sulfate, methane, ethane, and ethene were completed for the biowall process wells only.
1. During the 5R2008 event the water level in PT-17 was extremely low and water quality readings were not collected.
2. During the 11R2011 event, data was collected at MW-56 in October 2011.

Table 4
Chlorinated Organics in Groundwater
Round 21, June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Upgradient ↑

Sample Identification		Round	Sample Date	PCE (ug/L)	TCE (ug/L)	1,1-DCE (ug/L)	cis-DCE (ug/L)	trans-DCE (ug/L)	VC (ug/L)	1,1-DCA (ug/L)	1,2-DCA (ug/L)
		Class GA Standard (ug/L)		5	5	5	5	5	2	5	0.6
PT-18A	Upgradient of walls	1	3-Jan-07	1 U	2000	0.64 J	220	1.6	2.4	1 U	1 U
		2	17-Mar-07	1 U	1000	0.73 J	170	1.4	2.9	1 U	1 U
		3	5-Jun-07	1 U	1100	1.4	430	3.3	3.3	1 U	1 U
		4	15-Nov-07	1 U	2700	2.1	720	3.4	8.2	1 U	1 U
		5	24-Jun-08	1 U	220	1 U	200	0.9 J	1.4	1 U	1 U
		6	12-Dec-08	0.36 U	1400	1.3	510	2.4	4.6	0.75 U	0.21 U
		7	4-Jun-09	0.36 U	810 J	0.8 J	260	1.8	2.6	0.75 U	0.21 U
		8	17-Dec-09	1.5 U	2100	1.5 U	630	3.5 J	7.1	2 J	0.86 U
		9	1-Jul-10	0.15 U	120	0.11 U	28	0.2 U	0.18 U	0.25 U	0.1 U
		10	19-Dec-10	0.15 U	6.3	0.11 U	0.54 J	0.2 U	0.18 U	0.25 U	0.1 U
		11	22-Jul-11	1 U	0.13 U	1.5	15	0.2 U	120	62	0.1 U
		12	15-Dec-11	0.15 U	7.3	0.11 U	0.53 J	0.2 U	0.18 U	0.25 U	0.1 U
		13	21-Jun-12	13 J	3800	2.6	820	4.7	10	0.25 U	0.1 UJ
		14	12-Dec-12	0.15 U	8	0.11 U	0.8 J	0.2 U	0.18 U	0.25 U	0.1 U
		15	11-Jul-13	0.15 U	47	0.11 U	8.1	0.2 U	0.18 U	0.25 U	0.1 U
		16	13-Dec-13	0.15 U	9.4	0.11 U	1.4	0.2 U	0.18 U	0.25 U	0.1 U
		17	21-Jun-14	0.15 U	1200	0.77 J	240	1.2	2.2	0.25 U	0.1 U
		18	19-Dec-14	27	1800	2.2 U	420	5 J	3.6 U	5 U	2 U
		19	6-Jun-15	0.74 U	180 J	0.36 U	28	0.37 U	0.5 U	0.38 U	0.5 U
		20	18-Dec-15	0.74 U	160	0.36 U	31	0.37 U	0.5 U	0.38 U	0.5 U
		21	16-Jun-16	0.74 U	280	0.36 U	66	0.39 J	0.76 J	0.38 U	0.5 U
MWT-25	Upgradient of Biowall A	1	3-Jan-07	1 U	50	1 U	41	0.56 J	1.6	1 U	1 U
		2	17-Mar-07	1 U	55	1 U	84	1.2	9.6	1 U	1 U
		3	6-Jun-07	1 U	28	1 U	36	0.5 J	2.1	1 U	1 U
		4	15-Nov-07	1 U	26	1 U	17	1 U	0.64 J	1 U	1 U
		5	24-Jun-08	1 U	19	1 U	17	1 U	1 U	1 U	1 U
		6	15-Dec-08	0.36 U	3.2	0.29 U	0.63 J	0.13 U	0.24 U	0.75 U	0.21 U
		7	3-Jun-09	0.36 U	12	0.29 U	10	0.13 U	0.24 U	0.75 U	0.21 U
		8	17-Dec-09	0.36 U	4.2	0.38 U	3.3	0.42 U	0.24 U	0.29 U	0.21 U
		9	30-Jun-10	0.15 U	7.7	0.11 U	13	0.49 J	0.18 U	0.25 U	0.1 U
		10	19-Dec-10	0.15 U	1.9	0.11 U	0.97 J	0.2 U	0.18 U	0.25 U	0.1 U
		11	20-Jul-11	0.15 U	4.4	0.11 U	14	0.45 J	0.72 J	0.25 U	0.1 U
		12	15-Dec-11	0.15 U	1.6	0.11 U	0.30 J	0.20 U	0.18 U	0.25 U	0.1 U
		13	21-Jun-12	0.15 U	6.1	0.11 U	6.80	0.20 U	0.18 U	0.25 U	0.1 UJ
		14	12-Dec-12	0.15 U	1.3	0.11 U	0.39 J	0.20 U	0.18 U	0.25 U	0.1 U
		15	11-Jul-13	0.15 U	8.3	0.11 U	5.8	0.2 U	0.18 U	0.25 U	0.1 U
		16	13-Dec-13	0.15 U	4.6	0.11 U	3.3	0.2 U	0.47 J	0.25 U	0.1 U
		17	21-Jun-14	0.15 U	24	0.11 U	21	0.42 J	2.6	0.25 U	0.1 U
		18	19-Dec-14	0.15 U	2.5	0.11 U	1.7	0.2 U	0.18 U	0.25 U	0.1 U
		19	4-Jun-15	0.74 U	7.9 J	0.36 U	4.9	0.37 U	0.5 U	0.38 U	0.5 U
		20	18-Dec-15	0.74 U	2.6	0.36 U	1.7	0.37 U	0.5 U	0.38 U	0.5 U
		21	16-Jun-16	0.74 U	6.9	0.36 U	5.7	0.37 U	0.73 J	0.38 U	0.5 U
MWT-26	**Upgradient of Biowalls B1/B2**	1	3-Jan-07	1 U	10	1 U	19	0.6 J	2	1 U	1 U
		2	17-Mar-07	1 U	11	1 U	17	1	6.1	1 U	1 U
		3	5-Jun-07	1 U	3.2	1 U	11	0.7 J	4.4	1 U	1 U
		4	15-Nov-07	1 U	2.8	1 U	2.8	1 U	1 U	1 U	1 U
		5	24-Jun-08	1 U	1.7	1 U	3.3	1 U	1 U	1 U	1 U
		6	15-Dec-08	0.36 U	1.9	0.29 U	1	0.13 U	0.24 U	0.75 U	0.21 U
		7	3-Jun-09	0.36 U	3.6	0.29 U	6	0.13 U	3.5	0.75 U	0.21 U
		8	17-Dec-09	0.36 U	5.8	0.38 U	8.1	0.42 U	4.2	0.29 U	0.21 U
		9	29-Jun-10	0.15 U	1.7	0.11 U	5.5	0.37 J	0.18 U	0.25 U	0.1 U
		10	19-Dec-10	0.15 U	4.2	0.11 U	12	0.67 J	7.6	0.25 U	0.1 U
		11	20-Jul-11	0.15 U	1.6	0.11 U	9.8	0.81 J	4.4	0.25 U	0.1 U
		12	15-Dec-11	0.15 U	1.2	0.11 U	1.1	0.2 U	0.47 J	0.25 U	0.1 U
		13	20-Jun-12	0.15 U	1.6	0.11 U	4.4	0.24 J	1.1	0.25 U	0.1 UJ
		14	14-Dec-12	0.15 U	2.1	0.11 U	3.1	0.2 U	0.56 J	0.25 U	0.1 U
		15	11-Jul-13	0.15 U	2.1	0.11 U	5.8	0.2 U	1.6	0.25 U	0.1 U
		16	14-Dec-13	0.15 U	1.3	0.11 U	2.8	0.2 U	1	0.25 U	0.1 U
		17	19-Jun-14	0.15 U	0.83 J	0.11 U	4.5	0.4 J	1.1	0.25 U	0.1 U
		18	17-Dec-14	0.15 U	2.1	0.11 U	9.7	0.2 U	3.3	0.25 U	0.1 U
		19	4-Jun-15	0.74 U	1.3 J	0.36 U	5.4	0.49 J	0.86 J	0.38 U	0.5 U
		20	16-Dec-15	0.74 U	1.6	0.36 U	8.4	0.37 U	0.50 U	0.38 U	0.5 U
		21	15-Jun-16	0.74 U	2.1	0.36 U	3.7	0.37 U	1.2	0.38 U	0.5 U

↓ Downgradient

Table 4
Chlorinated Organics in Groundwater
Round 21, June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Upgradient ↓ Downgradient

Sample Identification		Round	Sample Date	PCE (ug/L)	TCE (ug/L)	1,1-DCE (ug/L)	cis-DCE (ug/L)	trans-DCE (ug/L)	VC (ug/L)	1,1-DCA (ug/L)	1,2-DCA (ug/L)
Class GA Standard (ug/L)				5	5	5	5	5	2	5	0.6
MWT-27	**In Biowall B1**	1	3-Jan-07	20 U	20 UJ	20 UJ	49 J	20 UJ	20 UJ	20 UJ	20 UJ
		2	16-Mar-07	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U
		3	5-Jun-07	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U
		4	15-Nov-07	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
		5	24-Jun-08	4 U	4 U	4 U	4 U	4 U	4 U	4 U	4 U
		6	15-Dec-08	3.6 U	1.8 U	2.9 U	1.6 U	1.3 U	2.4 U	7.5 U	2.1 U
		7	3-Jun-09	3.6 U	1.8 U	2.9 U	1.6 U	1.3 U	2.4 U	7.5 U	2.1 U
		8	16-Dec-09	1.8 U	2.3 U	1.9 U	1.9 U	2.1 U	3.1 J	1.5 U	1.1 U
		9	29-Jun-10	0.15 U	0.13 U	0.11 U	0.18 J	0.2 U	0.18 U	0.25 U	0.1 U
		10	20-Dec-10	0.15 U	0.51 J	0.11 U	1.1	0.2 U	2.1	0.25 U	0.1 U
		11	20-Jul-11	0.15 U	0.13 U	0.11 U	0.21 J	0.28 J	0.18 U	0.25 U	0.1 U
		12	14-Dec-11	0.15 UJ	0.13 U	0.11 U	1.4	0.2 U	3.0	0.25 U	0.1 U
		13	20-Jun-12	0.15 U	0.13 U	0.11 U	0.42 J	0.2 U	0.61 J	0.25 U	0.1 UJ
		14	13-Dec-12	0.15 U	0.13 U	0.11 U	0.15 U	0.2 U	0.18 U	0.25 U	0.1 U
		15	11-Jul-13	0.15 U	0.13 U	0.11 U	0.15 U	0.2 U	0.18 U	0.25 U	0.1 U
		16	12-Dec-13	0.15 U	0.13 U	0.11 U	0.48 J	0.2 U	0.84 J	0.25 U	0.1 U
		17	19-Jun-14	0.15 U	0.13 U	0.11 U	0.83 J	0.27 J	1	0.25 U	0.1 U
		18	17-Dec-14	0.15 U	0.13 U	0.11 U	0.70 J	0.2 U	1.2	0.25 U	0.1 U
		19	3-Jun-15	0.74 U	0.48 UJ	0.36 U	0.67 J	0.37 U	0.5 U	0.38 U	0.5 U
		20	16-Dec-15	0.74 U	0.48 U	0.36 U	0.87 J	0.37 U	0.5 U	0.38 U	0.5 U
		21	14-Jun-16	0.74 U	0.48 U	0.36 U	2.4	0.37 U	3.1	0.38 U	0.5 U
MWT-28	**In Biowall B2**	1	3-Jan-07	20 U	20 UJ	20 UJ	20 UJ	20 UJ	20 UJ	20 UJ	20 UJ
		2	16-Mar-07	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U
		3	5-Jun-07	20 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U
		4	15-Nov-07	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U
		5	25-Jun-08	4 U	4 U	4 U	4 U	4 U	4 U	4 U	4 U
		6	15-Dec-08	3.6 U	1.8 U	2.9 U	1.6 U	1.3 U	2.4 U	7.5 U	2.1 U
		7	3-Jun-09	0.36 U	0.18 U	0.29 U	0.16 U	0.13 U	0.24 U	0.75 U	0.21 U
		8	18-Dec-09	1.8 U	2.3 U	1.9 U	1.9 U	2.1 U	1.2 U	1.5 U	1.1 U
		9	29-Jun-10	0.15 U	0.13 U	0.11 U	0.15 U	0.2 U	0.18 U	0.25 U	0.1 U
		10	18-Dec-10	0.15 U	0.13 U	0.11 U	0.51 J	0.2 U	0.64 J	0.25 U	0.1 U
		11	19-Jul-11	0.15 U	0.13 U	0.11 U	0.15 U	0.2 U	0.18 U	0.25 U	0.1 U
		12	14-Dec-11	0.15 UJ	0.13 U	0.11 U	0.28 J	0.2 U	0.56 J	0.25 U	0.1 U
		13	20-Jun-12	0.15 U	0.13 U	0.11 U	0.15 U	0.2 U	0.18 U	0.25 U	0.1 UJ
		14	14-Dec-12	0.15 U	0.13 U	0.11 U	0.15 U	0.2 U	0.31 J	0.25 U	0.1 U
		15	11-Jul-13	0.15 U	0.13 U	0.11 U	0.15 U	0.2 U	0.18 U	0.25 U	0.1 U
		16	14-Dec-13	0.15 U	0.13 U	0.11 U	0.37 J	0.2 U	0.18 U	0.25 U	0.1 U
		17	19-Jun-14	0.15 U	0.13 U	0.11 U	0.15 U	0.2 U	0.18 U	0.25 U	0.1 U
		18	17-Dec-14	0.15 U	0.13 U	0.11 U	0.19 J	0.2 U	0.18 U	0.25 U	0.1 U
		19	3-Jun-15	0.74 U	0.48 UJ	0.36 U	0.41 U	0.37 U	0.5 U	0.38 U	0.5 U
		20	17-Dec-15	0.74 U	0.48 U	0.36 U	0.41 U	0.37 U	0.5 U	0.38 U	0.5 U
		21	14-Jun-16	0.74 U	0.48 U	0.36 U	0.41 U	0.37 U	0.5 U	0.38 U	0.5 U
MWT-29	**Downgradient of Biowall B2**	1	3-Jan-07	2 U	22	2 U	280	6.5	140	2 U	2 U
		2	16-Mar-07	4 U	19	4.5 U	220	7.75	165	4.5 U	5 U
		3	5-Jun-07	2 U	7.6	2 U	100	2.1	81	2 U	2 U
		4	14-Nov-07	1 U	4.4	1 U	96	0.83 J	74	1 U	1 U
		5	25-Jun-08	1 U	3.3	1 U	84	0.65 J	74	1 U	1 U
		6	15-Dec-08	0.36 U	6.6	0.29 U	91	0.6 J	80	0.75 U	0.21 U
		7	3-Jun-09	0.36 U	4.5	0.29 U	61	0.67 J	43	0.75 U	0.21 U
		8	16-Dec-09	0.36 U	3.5	0.38 U	37	0.65 J	29	0.29 U	0.21 U
		9	30-Jun-10	0.15 U	1.3	0.26 J	78	1.1	69	0.25 U	0.1 U
		10	19-Dec-10	0.15 U	2.1	0.4 J	38	0.77 J	27	0.25 U	0.1 U
		11	20-Jul-11	0.15 U	0.79 J	0.11 U	33	1.6	43	0.25 U	0.1 U
		12	14-Dec-11	0.15 UJ	2.4	0.11 U	8.5	0.26 J	5.9	0.25 U	0.1 U
		13	20-Jun-12	0.15 U	0.69 J	0.11 U	36	0.59 J	49	0.25 U	0.1 UJ
		14	14-Dec-12	0.15 U	3.3	0.11 U	25	0.44 J	11	0.25 U	0.1 U
		15	10-Jul-13	0.15 U	3.7	0.11 U	80	1.1	32	0.25 U	0.1 U
		16	12-Dec-13	0.15 U	2.1	0.11 U	28	0.42 J	20	0.25 U	0.1 U
		17	19-Jun-14	0.15 U	0.71 J	0.13 J	49	1.1	130	0.25 U	0.1 U
		18	17-Dec-14	0.15 U	2.3	0.11 U	18	0.2 U	7.5	0.25 U	0.1 U
		19	3-Jun-15	0.74 U	1.1 J	0.36 U	94	1.3	86	0.38 U	0.5 U
		20	17-Dec-15	0.74 U	2	0.36 U	35	0.43 J	45	0.38 U	0.5 U
		21	15-Jun-16	0.74 U	1.8	0.36 U	72	1.2	120	0.38 U	0.5 U

Table 4
Chlorinated Organics in Groundwater
Round 21, June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Upgradient ↑ Downgradient ↓

Sample Identification		Round	Sample Date	PCE (ug/L)	TCE (ug/L)	1,1-DCE (ug/L)	cis-DCE (ug/L)	trans-DCE (ug/L)	VC (ug/L)	1,1-DCA (ug/L)	1,2-DCA (ug/L)
		Class GA Standard (ug/L)		5	5	5	5	5	2	5	0.6
MWT-22	Downgradient of Biowall B2	1	3-Jan-07	2 U	5.2	2 U	130	2.7	98	2 U	2 U
		2	17-Mar-07	4 U	3.8 J	4 U	90	4 U	64	4 U	4 U
		3	6-Jun-07	1 U	6.5	1 U	120	3.2	81	1 U	1 U
		4	14-Nov-07	1 U	2.6	1 U	99	0.85 J	180	1 U	1 U
		5	25-Jun-08	5 U	3 J	5 U	68	5 U	42	5 U	5 U
		6	15-Dec-08	1.8 U	5.9	1.4 U	160	0.65 U	140	3.8 U	1 U
		7	3-Jun-09	0.36 U	2.2	0.29 U	66	0.77 J	89	0.75 U	0.21 U
		8	16-Dec-09	1.8 U	2.3 U	1.9 U	57	2.1 U	52	1.5 U	1.1 U
		9	1-Jul-10	0.15 U	0.6 J	0.12 J	41	1.3	57	0.25 U	0.1 U
		10	17-Dec-10	0.15 U	1.8	0.66 J	130	2.8	98	0.25 U	0.25 J
		11	20-Jul-11	0.15 U	0.32 J	0.11 U	23	2.0	59	0.25 U	0.1 U
		12	14-Dec-11	0.15 UJ	2.3	0.38 J	140	3.9	83	0.25 U	0.29 J
		13	21-Jun-12	0.15 U	0.48 J	0.11 U	57	5.0	90	0.25 U	0.1 UJ
		14	12-Dec-12	0.15 U	0.73 J	0.11 U	86	3.8	100	0.25 U	0.22 J
		15	10-Jul-13	0.15 U	2	0.27 J	150	6.2	84	0.25 U	0.28 J
		16	12-Dec-13	0.15 U	0.88 J	0.14 J	100	7.1	120	0.25 U	0.25 J
		17	21-Jun-14	0.15 U	0.19 J	0.11 U	19	2.8	65	0.25 U	0.11 J
		18	18-Dec-14	0.15 U	0.21 J	0.11 U	32	3.6	84	0.25 U	0.1 U
		19	5-Jun-15	0.74 U	0.48 UJ	0.36 U	32	4.0	81	0.38 U	0.5 U
		20	18-Dec-15	0.74 U	0.63 J	0.36 U	78	6.0	91	0.38 U	0.5 U
		21	16-Jun-16	0.74 U	0.54 J	0.36 U	39	5.6	110	0.38 U	0.5 U
PT-22	Between Biowalls B and C	1	3-Jan-07	1 U	11	1 U	57	0.86 J	22	1 U	3.3
		2	15-Mar-07	1 U	16	1 U	41	0.51 J	13	1 U	2.4
		3	5-Jun-07	1 U	8.5	1 U	61	0.72 J	32	1 U	5.6
		4	14-Nov-07	1 U	9.7	1 U	30	0.67 J	11	1 U	5
		5	26-Jun-08	1 U	4.1	1 U	26	0.57 J	13	1 U	3.9
		6	15-Dec-08	0.36 U	35	0.29 U	52	0.41 J	1.3	0.75 U	2.8
		7	2-Jun-09	0.36 U	6.9	0.29 U	41	0.81 J	11	0.75 U	4
		8	16-Dec-09	0.36 U	8.7	0.38 U	29	0.42 U	9.5	0.29 U	3
		9	30-Jun-10	0.15 U	4.6	0.11 U	43	0.75 J	11	0.25 U	3.2
		10	17-Dec-10	0.15 U	29	0.11 U	42	0.48 J	2.1	0.25 U	1.9
		11	22-Jul-11	0.15 U	31	0.11 U	42	0.2 U	0.18 U	0.25 U	0.1 U
		12	14-Dec-11	0.15 UJ	34	0.11 U	32	0.37 J	0.68 J	0.25 U	1.9
		13	21-Jun-12	0.15 U	7.9	0.11 U	31	0.84 J	4	0.25 U	2.1
		14	13-Dec-12	0.15 U	28	0.11 U	26	0.2 U	0.46 J	0.25 U	1.6
		15	9-Jul-13	0.15 U	38	0.11 U	49	0.45 J	1.6	0.25 U	2.3
		16	12-Dec-13	0.15 U	29	0.11 U	37	0.28 J	0.68 J	0.25 U	2
		17	21-Jun-14	0.15 U	23	0.11 U	52	1.3	2.9	0.25 U	3.1
		18	18-Dec-14	0.15 U	23	0.11 U	23	0.2 U	0.18 U	0.25 U	1.2
		19	6-Jun-15	0.74 U	34 J	0.36 U	33	0.37 U	0.5 U	0.38 U	2.3
		20	18-Dec-15	0.74 U	31	0.36 U	36	0.37 U	0.5 U	0.38 U	2.3
		21	16-Jun-16	0.74 U	23	0.36 U	44	0.87 J	2.8	0.38 U	2.6
MWT-23	**In Biowall C2**	1	3-Jan-07	4 U	4 U	4 U	60	4 U	23	4 U	2.3 J
		2	16-Mar-07	4 U	4 U	4 U	11	4 U	4.8	4 U	4 U
		3	6-Jun-07	2 U	2 U	2 U	3.1	2 U	2 U	2 U	1.6 J
		4	16-Nov-07	7 U	7 U	2.6 U	3.6 J	7 U	3.7 J	7 U	7 U
		5	25-Jun-08	1 U	1 U	1 U	1 U	1 U	1 U	1 U	0.6 J
		6	12-Dec-08	0.36 U	0.41 J	0.29 U	2.4	0.13 U	2.8	0.75 U	0.6 J
		7	2-Jun-09	0.36 U	0.18 U	0.29 U	0.42 U	0.13 U	0.24 U	0.75 U	0.64 J
		8	15-Dec-09	0.36 U	0.46 U	0.38 U	0.47 J	0.42 U	0.24 U	0.29 U	0.21 U
		9	29-Jun-10	0.15 U	0.13 U	0.11 U	0.41 J	0.2 U	0.18 U	0.25 U	0.66 J
		10	19-Dec-10	0.15 U	0.29 J	0.11 U	4.6	0.49 J	5.3	0.52 J	1.6
		11	19-Jul-11	0.15 U	0.13 U	0.11 U	0.57 J	0.22 J	0.33 J	0.25 U	1
		12	14-Dec-11	0.15 UJ	0.16 J	0.11 U	2.0	0.35 J	1.8	0.33 J	1.3
		13	20-Jun-12	0.15 U	0.13 U	0.11 U	0.55 J	0.42 J	0.33 J	0.25 U	0.65 J
		14	13-Dec-12	0.15 U	0.13 U	0.11 U	1.9	0.29 J	1.65	0.25 U	0.72 J
		15	10-Jul-13	0.15 U	0.13 U	0.11 U	3.3	1.4	2.9	0.5 J	1.2
		16	14-Dec-13	0.15 U	0.13 U	0.11 U	2.6	0.52 J	2.5	0.25 U	0.81 J
		17	20-Jun-14	0.14 J	0.13 U	0.11 U	0.45 J	0.47 J	0.37 J	0.43 J	0.66 J
		18	18-Dec-14	0.15 U	0.19 J	0.11 U	2.7	0.39 J	0.18 U	0.43 J	0.1 J
		19	4-Jun-15	0.74 U	0.48 UJ	0.36 U	1.0	1.2	0.5 U	0.38 U	0.9 J
		20	16-Dec-15	0.74 U	0.48 U	0.36 U	3.8	0.29 J	3.4	0.38 U	0.5 U
		21	15-Jun-16	0.74 UJ	0.48 U	0.36 U	1.2	0.54 J	1.9	0.38 U	1.3

Table 4
Chlorinated Organics in Groundwater
Round 21, June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Upgradient ↑ ↓ Downgradient

Sample Identification		Round	Sample Date	PCE (ug/L)	TCE (ug/L)	1,1-DCE (ug/L)	cis-DCE (ug/L)	trans-DCE (ug/L)	VC (ug/L)	1,1-DCA (ug/L)	1,2-DCA (ug/L)
		Class GA Standard (ug/L)		5	5	5	5	5	2	5	0.6
MWT-24	Downgradient of Biowalls C1/C2	1	3-Jan-07	1 U	0.94 J	1 U	210	2.1	19	0.81 J	1 U
		2	15-Mar-07	1 U	1 U	1 U	68	0.88 J	45	0.83 J	1 U
		3	5-Jun-07	2 U	2 U	2 U	19	2 U	22	1.1 J	2 U
		4	13-Nov-07	1 U	1.6	1 U	6.7	1 U	3.8	1 U	1 U
		5	26-Jun-08	5 U	5 U	5 U	31	5 U	5 U	5 U	5 U
		6	12-Dec-08	0.36 U	6	0.29 U	52	0.13 U	3.6	0.75 U	0.21 U
		7	2-Jun-09	0.36 U	4.8	0.29 U	38	0.13 U	7.3	0.75 U	0.21 U
		8	15-Dec-09	0.36 U	4.7	0.7 J	32	0.42 U	4	0.29 U	0.21 U
		9	1-Jul-10	0.15 U	5	0.11 U	31	0.41 J	7.5	0.79 J	0.1 U
		10	17-Dec-10	0.15 U	3.3	0.11 U	23	1	4.3	0.58 J	0.1 U
		11	21-Jul-11	0.15 U	5.6	0.11 U	39	1.6	17	0.25 U	3.3
		12	13-Dec-11	0.15 U	3.1	0.11 U	16	0.39 J	2.3	0.44 J	0.1 U
		13	19-Jun-12	0.15 U	2.7	0.11 U	28	1.5	5.3	0.8 J	0.1 UJ
		14	12-Dec-12	0.15 U	4.1	0.11 U	25	0.2 U	0.31 J	0.57 J	0.1 U
		15	9-Jul-13	0.15 U	3.7	0.11 U	24	1.2	2.1	0.7 J	0.1 U
		16	11-Dec-13	0.15 U	1.9	0.11 U	21	1.5	2.4	0.67 J	0.1 U
		17	21-Jun-14	0.15 U	1.5	0.11 U	21	1.6	3.6	0.25 U	0.1 U
		18	18-Dec-14	0.15 U	1.9	0.11 U	11	0.2 U	0.18 U	0.38 J	0.1 U
		19	5-Jun-15	0.74 U	4.0 J	0.36 U	16	0.74 J	1.1	0.58 J	0.5 U
		20	18-Dec-15	0.74 U	3.0	0.36 U	18	1.1	2.4	0.62 J	0.5 U
		21	16-Jun-16	0.74 U	1.8	0.36 U	15	1.7	3.2	0.53 J	0.5 U
PT-17	Downgradient of biowalls	1	2-Jan-07	1 U	6	1 U	62	1 U	21	1 U	1 U
		2	15-Mar-07	2 U	11	2 U	26	2 U	21	2 U	2 U
		3	5-Jun-07	1 U	3.4	1 U	43	0.77 J	9.9	1 U	1 U
		4	13-Nov-07	1 U	15	1 U	27	0.54 J	22	1 U	1 U
		5	26-Jun-08	1 U	8.5	1 U	21	1 U	23	1 U	1 U
		6	11-Dec-08	0.36 U	9.2	0.29 U	24	0.46 J	10	0.75 U	0.21 U
		7	2-Jun-09	0.36 U	8	0.29 U	56	1.1	55	0.75 U	0.21 U
		8	15-Dec-09	0.36 U	7.8	0.38 U	65	1.8	20	0.29 U	0.21 U
		9	1-Jul-10	0.15 U	3	0.24 J	81	3.2	53	0.25 U	0.1 U
		10	18-Dec-10	0.15 U	8.1	0.42 J	39	2.2	16	0.25 U	0.1 U
		11	21-Jul-11	1 U	4.5	0.11 U	94	7.0	56	0.25 UJ	0.1 U
		12	13-Dec-11	0.15 U	11	0.11 U	25	1.8	12	0.25 U	0.1 U
		13	19-Jun-12	0.15 U	6.9	0.37 J	170	18.0	66	0.25 U	0.1 UJ
		14	13-Dec-12	0.15 U	12	0.18 J	68	8.3	21	0.25 U	0.1 U
		15	10-Jul-13	0.15 U	14	0.11 U	38	5.2	7.9	0.25 U	0.1 U
		16	13-Dec-13	0.15 U	8.4	0.16 J	64	11	17	0.25 U	0.1 U
		17	20-Jun-14	0.15 U	3.4	0.32 J	130	18	55	0.25 U	0.1 U
		18	16-Dec-14	0.15 U	7.4	0.31 J	120	22	38	0.25 U	0.1 U
		19	5-Jun-15	0.74 U	9.0 J	0.36 U	57	13	15	0.38 U	0.5 U
		20	17-Dec-15	0.74 U	13	0.36 U	27	4.4	0.5 U	0.38 U	0.5 U
		21	15-Jun-16	0.74 U	5.4	0.36 U	61	11	25	0.38 U	0.5 U
MWT-7	Immediately upgradient of ZVI wall	1	4-Jan-07	1 U	490	1 U	35	1 U	0.51 J	1 U	1 U
		2	15-Mar-07	1 U	440	1 U	42	1 U	9.7	1 U	1 U
		3	5-Jun-07	1 U	410	1 U	61	1 U	18	1 U	1 U
		4	13-Nov-07	1 U	510	1 U	90	1 U	24	1 U	1 U
		5	25-Jun-08	1 U	440	1 U	90	1 U	12	1 U	1 U
		6	15-Dec-08	0.36 U	410	0.29 U	79	0.13 U	13	0.75 U	0.21 U
		7	2-Jun-09	0.36 U	330	0.29 U	68	0.13 U	9.3	0.75 U	0.21 U
		8	15-Dec-09	0.36 U	350	0.38 U	140	0.55 J	21	0.48 J	0.21 U
		9	1-Jul-10	0.15 U	330	0.78 J	170	0.91 J	15	0.25 U	0.1 U
		10	18-Dec-10	0.15 U	310	0.98 J	120	0.75 J	15	0.25 U	0.1 U
		11	22-Jul-11	0.15 U	0.52 J	0.11 U	12	0.34 J	2.6	0.94 J	0.1 U
		12	13-Dec-11	0.15 U	2.3	0.11 U	56	0.24 J	4.3	1.2	0.1 U
		13	19-Jun-12	0.15 U	280	0.59 J	140	0.64 J	11	0.25 U	0.1 UJ
		14	13-Dec-12	0.15 U	280	0.5 J	100	0.33 J	5.9	0.25 U	0.1 U
		15	10-Jul-13	0.15 U	300	0.5 J	110	0.46 J	2.6	0.25 U	0.1 U
		16	13-Dec-13	0.3 U	370	0.22 U	140	0.4 U	9.6	0.5 U	0.2 U
		17	20-Jun-14	0.15 U	190	0.69 J	110	0.73 J	9.6	0.25 U	0.1 U
		18	16-Dec-14	0.75 U	260	1.8 J	150	1.8 J	16	1.3 U	0.5 U
		19	5-Jun-15	0.74 U	200 J	0.63 J	100	0.57 J	6.1	0.38 U	0.5 U
		20	17-Dec-15	0.74 U	170	0.36 U	150	0.9 J	11	0.38 U	0.5 U
		21	15-Jun-16	0.74 U	170	0.51 J	95	0.5 J	5.5	0.38 U	0.5 U

Table 4
Chlorinated Organics in Groundwater
Round 21, June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Upgradient ↓ Downgradient

Sample Identification		Round	Sample Date	PCE (ug/L)	TCE (ug/L)	1,1-DCE (ug/L)	cis-DCE (ug/L)	trans-DCE (ug/L)	VC (ug/L)	1,1-DCA (ug/L)	1,2-DCA (ug/L)
		Class GA Standard (ug/L)		5	5	5	5	5	2	5	0.6
PT-24	Downgradient of ZVI wall	1	2-Jan-07	1 U	4	1 U	54	0.86 J	0.6 J	0.68 J	1 U
		2	15-Mar-07	1 U	2.8	1 U	38	0.81 J	1 U	1 U	1 U
		3	5-Jun-07	1 U	3.1	1 U	60	1.6	2.6	0.75 J	1 U
		4	13-Nov-07	1 U	3.8	1 U	39	1 U	1 U	0.56 J	1 U
		5	26-Jun-08	1 U	2.4	1 U	48	1.1	1.9	0.69 J	1 U
		6	12-Dec-08	0.36 U	2.2	0.29 U	34	0.36 J	0.26 J	0.75 U	0.21 U
		7	2-Jun-09	0.36 U	1.7	0.29 U	32	0.83 J	2	0.75 U	0.21 U
		8	15-Dec-09	0.36 U	1.7	0.38 U	28	0.61 J	1.6	0.29 U	0.21 U
		9	30-Jun-10	0.15 U	0.39 J	0.11 U	33	1.1	3.8	0.54 J	0.1 U
		10	17-Dec-10	0.15 U	0.53 J	0.11 U	30	1.4	7.7	0.54 J	0.1 U
		11	21-Jul-11	0.15 U	0.38 J	0.11 U	37	1.4	7.9	0.78 J	0.1 U
		12	13-Dec-11	0.15 U	0.82 J	0.11 U	21	0.63 J	2.9	0.48 J	0.1 U
		13	19-Jun-12	0.15 U	0.87 J	0.11 U	30	0.84 J	2.8	0.57 J	0.1 UJ
		14	12-Dec-12	0.15 U	1.1	0.11 U	18	0.38 J	0.18 U	0.32 J	0.1 U
		15	9-Jul-13	0.15 U	1.6	0.11 U	24	0.8 J	0.83 J	0.51 J	0.1 U
		16	11-Dec-13	0.15 U	1.3	0.11 U	23	0.86 J	1.8	0.52 J	0.1 U
		17	20-Jun-14	0.15 U	1.3	0.11 U	23	1	1.7	0.25 U	0.1 U
		18	19-Dec-14	0.15 U	0.85	0.11 U	13	0.53 J	0.18 U	0.29 J	0.1 U
		19	6-Jun-15	0.74 U	0.48 J	0.36 U	18	1.2	2.1	0.41 J	0.5 U
		20	18-Dec-15	0.74 U	0.74 J	0.36 U	18	0.75 J	1.2	0.38 U	0.5 U
		21	15-Jun-16	0.74 U	0.48 U	0.36 U	18	1.1	3.6	0.42 J	0.5 U
MW-56	Off-site well	1	4-Jan-07	1 U	1 U	1 U	1.2	1 U	1 U	1 U	1 U
		3	6-Jun-07	1 U	1 U	1 U	1.7	1 U	1 U	1 U	1 U
		5	26-Jun-08	1 U	1 U	1 U	1.3	1 U	1 U	1 U	1 U
		6	11-Dec-08	0.36 U	0.33 J	0.29 U	0.4 J	0.13 U	0.24 U	0.75 U	0.21 U
		7	4-Jun-09	0.36 U	0.18 U	0.29 U	1	0.13 U	0.24 U	0.75 U	0.21 U
		8	18-Dec-09	0.36 U	0.46 U	0.38 U	0.56 J	0.42 U	0.24 U	0.29 U	0.21 U
		9	1-Jul-10	0.15 U	0.13 U	0.11 U	0.61 J	0.2 U	0.18 U	0.25 U	0.1 U
		10	19-Dec-10	0.15 U	0.13 U	0.11 U	0.86 J	0.2 U	0.18 U	0.25 U	0.1 U
		11	4-Oct-11	0.15 U	0.13 U	0.11 U	2.3	0.2 U	0.18 U	0.25 U	0.1 U
		12	12-Dec-11	0.15 U	0.13 U	0.11 U	0.95 J	0.2 U	0.18 U	0.25 U	0.1 U
		13	18-Jun-12	0.15 U	0.13 U	0.11 U	2.2	0.2 U	0.18 U	0.25 U	0.1 UJ
		14	14-Dec-12	0.15 U	0.13 U	0.11 U	0.85 J	0.2 U	0.18 U	0.25 U	0.1 U
		15	9-Jul-13	0.15 U	0.13 U	0.11 U	2.2	0.2 U	0.18 U	0.25 U	0.1 U
		16	11-Dec-13	0.15 U	0.13 U	0.11 U	1.7	0.2 U	0.18 U	0.25 U	0.1 U
		17	22-Jun-14	0.15 U	0.13 U	0.11 U	0.98 J	0.2 U	0.18 U	0.25 U	0.1 U
		18	19-Dec-14	0.15 U	0.13 U	0.11 U	0.89 J	0.2 U	0.18 U	0.25 U	0.1 U
		19	6-Jun-15	0.74 U	0.48 UJ	0.36 U	1.1	0.37 U	0.5 U	0.38 U	0.5 U
		20	19-Dec-15	0.74 U	0.48 U	0.36 U	1.4	0.37 U	0.5 U	0.38 U	0.5 U
		21	17-Jun-16	0.74 U	0.48 U	0.36 U	2.8	0.37 U	0.5 U	0.38 U	0.5 U

Notes:

1. Sample duplicate pairs were collected at MWT-28 in Jan-07, June-09, June-10, June-12, Dec-13, and June-15; MWT-29 in Mar-07 and Jun-08; MWT-27 in Jun-07, Dec-08, Dec-09, July-11, July-13, Dec-14, June-16; and MWT-23 in Nov-07, Dec-10, Dec-11, Dec-12, June-14, Dec-15. If an analyte was detected in the sample, but not detected in the duplicate (or vice versa), the non-detect value was taken at half the detection limit averaged with the detect value.
2. Wells in bold are the biowall process monitoring wells.
3. Grey shading indicates that the concentration was detected above its Class GA groundwater standard. The Class GA Groundwater standard for TCE and cis-DCE is 5 ug/L; for VC the Class GA standard is 2 ug/L.

U = compound was not detected; detection limit shown.
J = the reported value is an estimated concentration.
UJ = the compound was not detected; the associated reporting limit is approximate.

Table 5
Summary of VOCs in Groundwater
Round 21 - June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Area									ASH LANDFILL		ASH LANDFILL		ASH LANDFILL		ASH LANDFILL		ASH LANDFILL		ASH LANDFILL	
Loc ID									PT-18A		MWT-25		MWT-26		MWT-27		MWT-27		MWT-28	
Matrix									GW		GW		GW		GW		GW		GW	
Sample ID									ALBW20360		ALBW20366		ALBW20369		ALBW20370		ALBW20371		ALBW20372	
Sample Date									6/16/2016		6/16/2016		6/15/2016		6/14/2016		6/14/2016		6/14/2016	
QC Type									SA		SA		SA		SA		DU		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									21		21		21		21		21		21	
Filtered									Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Volatile Organic Compounds																				
1,1-Dichloroethane	UG/L	0.53	13%	GA	5	0	2	15	0.38	U	0.38	U	0.38	U	0.38	U	0.38	U	0.38	U
1,1-Dichloroethene	UG/L	0.51	7%	GA	5	0	1	15	0.36	U	0.36	U	0.36	U	0.36	U	0.36	U	0.36	U
1,2-Dichloroethane	UG/L	2.6	13%	GA	0.6	2	2	15	0.5	U	0.5	U	0.5	U	0.5	U	0.5	U	0.5	U
Chloroform	UG/L	2.3	7%	GA	7	0	1	15	2.3		0.5	U	0.5	U	0.5	U	0.5	U	0.5	U
Cis-1,2-Dichloroethene	UG/L	95	93%	GA	5	9	14	15	**66**		**5.7**		3.7		2.3		2.5		0.41	U
Trans-1,2-Dichloroethene	UG/L	11	60%	GA	5	2	9	15	0.39	J	0.37	U	0.37	U	0.37	U	0.37	U	0.37	U
Trichloroethene	UG/L	280	60%	GA	5	5	9	15	**280**		**6.9**		2.1		0.48	U	0.48	U	0.48	U
Vinyl chloride	UG/L	120	87%	GA	2	9	13	15	0.76	J	0.73	J	1.2		**2.7**		**3.5**		0.5	U

Notes:
1. Only detected VOCs are included in this summary table.
2. The cleanup goal values are NYSDEC Class GA GW Standards (TOGS 1.1.1, June 1998).
3. Shading indicates a concentration above the GA GW Standard.

U = compound was not detected
J = the reported value is an estimated concentration

Table 5
Summary of VOCs in Groundwater
Round 21 - June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Area									ASH LANDFILL		ASH LANDFILL		ASH LANDFILL		ASH LANDFILL		ASH LANDFILL		ASH LANDFILL	
Loc ID									MWT-29		MWT-22		PT-22		MWT-23		MWT-24		PT-17	
Matrix									GW		GW		GW		GW		GW		GW	
Sample ID									ALBW20373		ALBW20364		ALBW20361		ALBW20368		ALBW20365		ALBW20359	
Sample Date									6/15/2016		6/16/2016		6/16/2016		6/15/2016		6/16/2016		6/15/2016	
QC Type									SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									21		21		21		21		21		21	
Filtered									Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Volatile Organic Compounds																				
1,1-Dichloroethane	UG/L	0.53	13%	GA	5	0	2	15	0.38	U	0.38	U	0.38	U	0.38	U	0.53	J	0.38	U
1,1-Dichloroethene	UG/L	0.51	7%	GA	5	0	1	15	0.36	U	0.36	U	0.36	U	0.36	U	0.36	U	0.36	U
1,2-Dichloroethane	UG/L	2.6	13%	GA	0.6	2	2	15	0.5	U	0.5	U	**2.6**		**1.3**		0.5	U	0.5	U
Chloroform	UG/L	2.3	7%	GA	7	0	1	15	0.5	U	0.5	U	0.5	U	0.5	U	0.5	U	0.5	U
Cis-1,2-Dichloroethene	UG/L	95	93%	GA	5	9	14	15	**72**		**39**		**44**		1.2		**15**		**61**	
Trans-1,2-Dichloroethene	UG/L	11	60%	GA	5	2	9	15	1.2		**5.6**		0.87	J	0.54	J	1.7		**11**	
Trichloroethene	UG/L	280	60%	GA	5	5	9	15	1.8		0.54	J	**23**		0.48	U	1.8		**5.4**	
Vinyl chloride	UG/L	120	87%	GA	2	9	13	15	**120**		**110**		**2.8**		1.9		**3.2**		**25**	

Notes:
1. Only detected VOCs are included in this summary table.
2. The cleanup goal values are NYSDEC Class GA GW Standards (TOGS 1.1.1, June 1998).
3. Shading indicates a concentration above the GA GW Standard.

U = compound was not detected
J = the reported value is an estimated concentration

Table 5
Summary of VOCs in Groundwater
Round 21 - June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity

Area									ASH LANDFILL		ASH LANDFILL		ASH LANDFILL	
Loc ID									MWT-7		PT-24		MW-56	
Matrix									GW		GW		GW	
Sample ID									ALBW20363		ALBW20362		ALBW20367	
Sample Date									6/15/2016		6/15/2016		6/17/2016	
QC Type									SA		SA		SA	
Study ID									LTM		LTM		LTM	
Sample Round									21		21		21	
Filtered									Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual
Volatile Organic Compounds														
1,1-Dichloroethane	UG/L	0.53	13%	GA	5	0	2	15	0.38	U	0.42	J	0.38	U
1,1-Dichloroethene	UG/L	0.51	7%	GA	5	0	1	15	0.51	J	0.36	U	0.36	U
1,2-Dichloroethane	UG/L	2.6	13%	GA	0.6	2	2	15	0.5	U	0.5	U	0.5	U
Chloroform	UG/L	2.3	7%	GA	7	0	1	15	0.5	U	0.5	U	0.5	U
Cis-1,2-Dichloroethene	UG/L	95	93%	GA	5	9	14	15	**95**		**18**		2.8	
Trans-1,2-Dichloroethene	UG/L	11	60%	GA	5	2	9	15	0.5	J	1.1		0.37	U
Trichloroethene	UG/L	280	60%	GA	5	5	9	15	**170**		0.48	U	0.48	U
Vinyl chloride	UG/L	120	87%	GA	2	9	13	15	**5.5**		**3.6**		0.5	U

Notes:
1. Only detected VOCs are included in this summary table.
2. The cleanup goal values are NYSDEC Class GA GW Standards (TOGS 1.1.1, June 1998).
3. Shading indicates a concentration above the GA GW Standard.

U = compound was not detected
J = the reported value is an estimated concentration

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\ASH LANDFILL LTM\ROUND 21 - JUNE 2016\R21 LETTER REPORT\DRAFT\, DATE: 07/07/2016 12:54:09PM, P0091241

LEGEND:

- PAVED ROAD
- DIRT ROAD
- GROUND CONTOUR AND ELEVATION (650)
- TREE
- WETLAND & DESIGNATION (W-B)
- BRUSH
- CHAIN LINK FENCE
- UTILITY POLE
- APPROXIMATE LOCATION OF FIRE HYDRANT
- FUEL OR UNDERGROUND STORAGE TANK
- SURVEY MONUMENT (SEAD-1 EL.630.90')
- MONITORING WELL AND DESIGNATION (PT-22)
- RAILROAD TRACKS
- WATER MAIN
- POST CONSTRUCTION AS BUILT GROUND ELEVATION CONTOUR (670)
- PILOT STUDY BIOWALL (2005)
- SINGLE BIOWALL (2006)
- DOUBLE-WIDE BIOWALL (2006)
- ZERO VALENT IRON WALL (1998)
- LIMITS OF LANDFILL
- SEDA PROPERTY BOUNDARY

100 0 100 200
(feet)

PARSONS

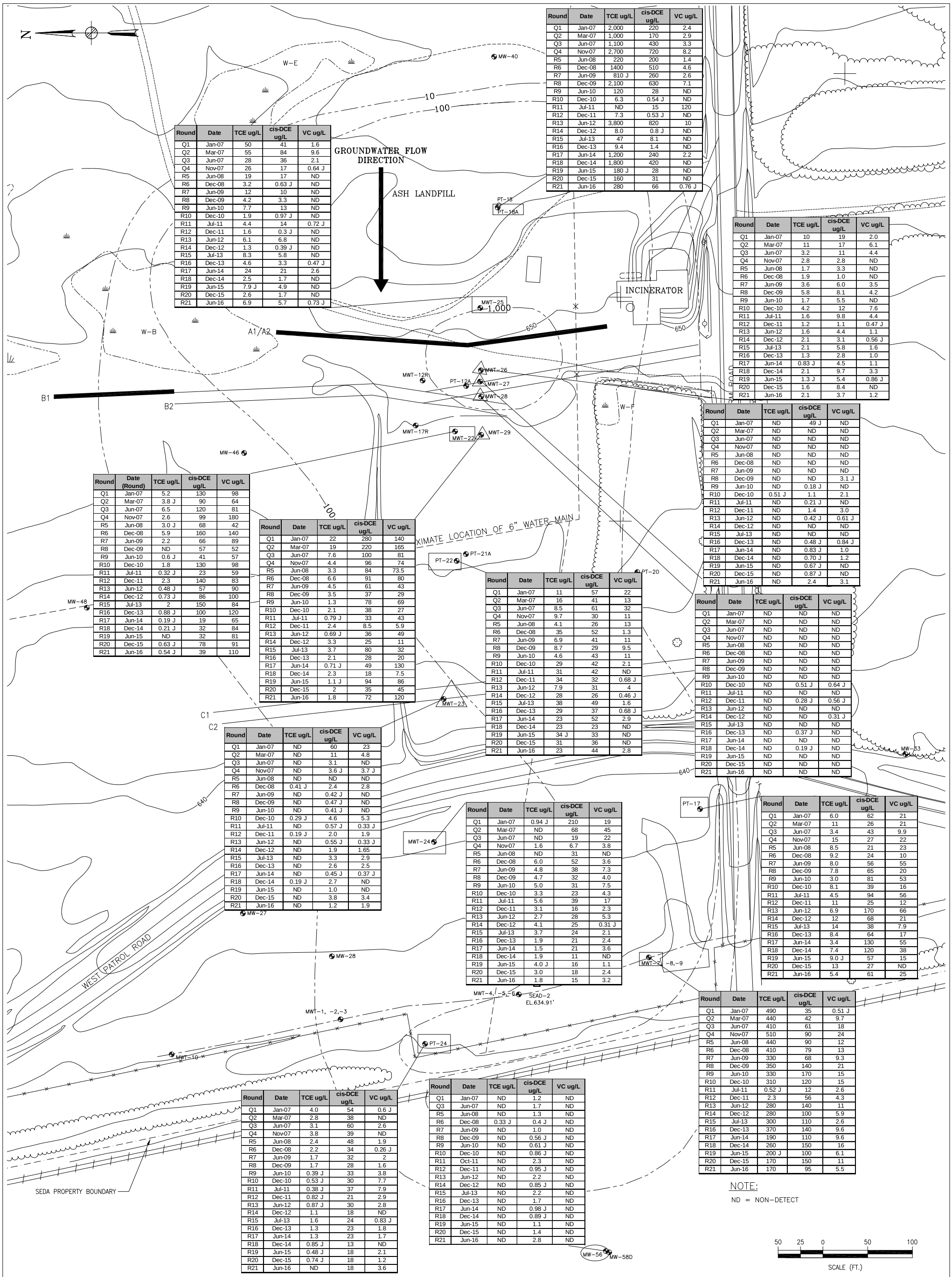
CLIENT/PROJECT TITLE

SENECA ARMY DEPOT
ASH LANDFILL
LONG TERM MONITORING REPORT

DEPT. ENVIRONMENTAL ENGINEERING | Dwg. No.

FIGURE 1
ASH LANDFILL
SITE PLAN

SCALE | DATE JULY 2016 | REV –



Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	2,000	220	2.4
Q2	Mar-07	1,000	170	2.9
Q3	Jun-07	1,100	430	3.3
Q4	Nov-07	2,700	720	8.2
R5	Jun-08	220	200	1.4
R6	Dec-08	1400	510	4.6
R7	Jun-09	810 J	260	2.6
R8	Dec-09	2,100	630	7.1
R9	Jun-10	120	28	ND
R10	Dec-10	6.3	0.54 J	ND
R11	Jul-11	ND	15	120
R12	Dec-11	7.3	0.53 J	ND
R13	Jun-12	3,800	820	10
R14	Dec-12	8.0	0.8 J	ND
R15	Jul-13	47	8.1	ND
R16	Dec-13	9.4	1.4	ND
R17	Jun-14	1,200	240	2.2
R18	Dec-14	1,800	420	ND
R19	Jun-15	180 J	28	ND
R20	Dec-15	160	31	ND
R21	Jun-16	280	66	0.76 J

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	50	41	1.6
Q2	Mar-07	55	84	9.6
Q3	Jun-07	28	36	2.1
Q4	Nov-07	26	17	0.64 J
R5	Jun-08	19	17	ND
R6	Dec-08	3.2	0.63 J	ND
R7	Jun-09	12	10	ND
R8	Dec-09	4.2	3.3	ND
R9	Jun-10	7.7	13	ND
R10	Dec-10	1.9	0.97 J	ND
R11	Jul-11	4.4	14	0.72 J
R12	Dec-11	1.6	0.3 J	ND
R13	Jun-12	6.1	6.8	ND
R14	Dec-12	1.3	0.39 J	ND
R15	Jul-13	8.3	5.8	ND
R16	Dec-13	4.6	3.3	0.47 J
R17	Jun-14	24	21	2.6
R18	Dec-14	2.5	1.7	ND
R19	Jun-15	7.9 J	4.9	ND
R20	Dec-15	2.6	1.7	ND
R21	Jun-16	6.9	5.7	0.73 J

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	10	19	2.0
Q2	Mar-07	11	17	6.1
Q3	Jun-07	3.2	11	4.4
Q4	Nov-07	2.8	2.8	ND
R5	Jun-08	1.7	3.3	ND
R6	Dec-08	1.9	1.0	ND
R7	Jun-09	3.6	6.0	3.5
R8	Dec-09	5.8	8.1	4.2
R9	Jun-10	1.7	5.5	ND
R10	Dec-10	4.2	12	7.6
R11	Jul-11	1.6	9.8	4.4
R12	Dec-11	1.2	1.1	0.47 J
R13	Jun-12	1.6	4.4	1.1
R14	Dec-12	2.1	3.1	0.56 J
R15	Jul-13	2.1	5.8	1.6
R16	Dec-13	1.3	2.8	1.0
R17	Jun-14	0.83 J	4.5	1.1
R18	Dec-14	2.1	9.7	3.3
R19	Jun-15	1.3 J	5.4	0.86 J
R20	Dec-15	1.6	8.4	ND
R21	Jun-16	2.1	3.7	1.2

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	ND	49 J	ND
Q2	Mar-07	ND	ND	ND
Q3	Jun-07	ND	ND	ND
Q4	Nov-07	ND	ND	ND
R5	Jun-08	ND	ND	ND
R6	Dec-08	ND	ND	ND
R7	Jun-09	ND	ND	ND
R8	Dec-09	ND	ND	3.1 J
R9	Jun-10	ND	0.18 J	ND
R10	Dec-10	0.51 J	1.1	2.1
R11	Jul-11	ND	0.21 J	ND
R12	Dec-11	ND	1.4	3.0
R13	Jun-12	ND	0.42 J	0.61 J
R14	Dec-12	ND	ND	ND
R15	Jul-13	ND	ND	ND
R16	Dec-13	ND	0.48 J	0.84 J
R17	Jun-14	ND	0.83 J	1.0
R18	Dec-14	ND	0.70 J	1.2
R19	Jun-15	ND	0.67 J	ND
R20	Dec-15	ND	0.87 J	ND
R21	Jun-16	ND	2.4	3.1

Round	Date (Round)	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	5.2	130	98
Q2	Mar-07	3.8 J	90	64
Q3	Jun-07	6.5	120	81
Q4	Nov-07	2.6	99	180
R5	Jun-08	3.0 J	68	42
R6	Dec-08	5.9	160	140
R7	Jun-09	2.2	66	89
R8	Dec-09	ND	57	52
R9	Jun-10	0.6 J	41	57
R10	Dec-10	1.8	130	98
R11	Jul-11	0.32 J	23	59
R12	Dec-11	2.3	140	83
R13	Jun-12	0.48 J	57	90
R14	Dec-12	0.73 J	86	100
R15	Jul-13	2	150	84
R16	Dec-13	0.88 J	100	120
R17	Jun-14	0.19 J	19	65
R18	Dec-14	0.21 J	32	84
R19	Jun-15	ND	32	81
R20	Dec-15	0.63 J	78	91
R21	Jun-16	0.54 J	39	110

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	22	280	140
Q2	Mar-07	19	220	165
Q3	Jun-07	7.6	100	81
Q4	Nov-07	4.4	96	74
R5	Jun-08	3.3	84	73.5
R6	Dec-08	6.6	91	80
R7	Jun-09	4.5	61	43
R8	Dec-09	3.5	37	29
R9	Jun-10	1.3	78	69
R10	Dec-10	2.1	38	27
R11	Jul-11	0.79 J	33	43
R12	Dec-11	2.4	8.5	5.9
R13	Jun-12	0.69 J	36	49
R14	Dec-12	3.3	25	11
R15	Jul-13	3.7	80	32
R16	Dec-13	2.1	28	20
R17	Jun-14	0.71 J	49	130
R18	Dec-14	2.3	18	7.5
R19	Jun-15	1.1 J	94	86
R20	Dec-15	2	35	45
R21	Jun-16	1.8	72	120

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	11	57	22
Q2	Mar-07	16	41	13
Q3	Jun-07	8.5	61	32
Q4	Nov-07	9.7	30	11
R5	Jun-08	4.1	26	13
R6	Dec-08	35	52	1.3
R7	Jun-09	6.9	41	11
R8	Dec-09	8.7	29	9.5
R9	Jun-10	4.6	43	11
R10	Dec-10	29	42	2.1
R11	Jul-11	31	42	ND
R12	Dec-11	34	32	0.68 J
R13	Jun-12	7.9	31	4
R14	Dec-12	28	26	0.46 J
R15	Jul-13	38	49	1.6
R16	Dec-13	29	37	0.68 J
R17	Jun-14	23	52	2.9
R18	Dec-14	23	23	ND
R19	Jun-15	34 J	33	ND
R20	Dec-15	31	36	ND
R21	Jun-16	23	44	2.8

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	ND	ND	ND
Q2	Mar-07	ND	ND	ND
Q3	Jun-07	ND	ND	ND
Q4	Nov-07	ND	ND	ND
R5	Jun-08	ND	ND	ND
R6	Dec-08	ND	ND	ND
R7	Jun-09	ND	ND	ND
R8	Dec-09	ND	ND	ND
R9	Jun-10	ND	ND	ND
R10	Dec-10	ND	0.51 J	0.64 J
R11	Jul-11	ND	ND	ND
R12	Dec-11	ND	0.28 J	0.56 J
R13	Jun-12	ND	ND	ND
R14	Dec-12	ND	ND	0.31 J
R15	Jul-13	ND	ND	ND
R16	Dec-13	ND	0.37 J	ND
R17	Jun-14	ND	ND	ND
R18	Dec-14	ND	0.19 J	ND
R19	Jun-15	ND	ND	ND
R20	Dec-15	ND	ND	ND
R21	Jun-16	ND	ND	ND

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	ND	60	23
Q2	Mar-07	ND	11	4.8
Q3	Jun-07	ND	3.1	ND
Q4	Nov-07	ND	3.6 J	3.7 J
R5	Jun-08	ND	ND	ND
R6	Dec-08	0.41 J	2.4	2.8
R7	Jun-09	ND	0.42 J	ND
R8	Dec-09	ND	0.47 J	ND
R9	Jun-10	ND	0.41 J	ND
R10	Dec-10	0.29 J	4.6	5.3
R11	Jul-11	ND	0.57 J	0.33 J
R12	Dec-11	0.19 J	2.0	1.9
R13	Jun-12	ND	0.55 J	0.33 J
R14	Dec-12	ND	1.9	1.65
R15	Jul-13	ND	3.3	2.9
R16	Dec-13	ND	2.6	2.5
R17	Jun-14	ND	0.45 J	0.37 J
R18	Dec-14	0.19 J	2.7	ND
R19	Jun-15	ND	1.0	ND
R20	Dec-15	ND	3.8	3.4
R21	Jun-16	ND	1.2	1.9

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	0.94 J	210	19
Q2	Mar-07	ND	68	45
Q3	Jun-07	ND	19	22
Q4	Nov-07	1.6	6.7	3.8
R5	Jun-08	ND	31	ND
R6	Dec-08	6.0	52	3.6
R7	Jun-09	4.8	38	7.3
R8	Dec-09	4.7	32	4.0
R9	Jun-10	5.0	31	7.5
R10	Dec-10	3.3	23	4.3
R11	Jul-11	5.6	39	17
R12	Dec-11	3.1	16	2.3
R13	Jun-12	2.7	28	5.3
R14	Dec-12	4.1	25	0.31 J
R15	Jul-13	3.7	24	2.1
R16	Dec-13	1.9	21	2.4
R17	Jun-14	1.5	21	3.6
R18	Dec-14	1.9	11	ND
R19	Jun-15	4.0 J	16	1.1
R20	Dec-15	3.0	18	2.4
R21	Jun-16	1.8	15	3.2

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	6.0	62	21
Q2	Mar-07	11	26	21
Q3	Jun-07	3.4	43	9.9
Q4	Nov-07	15	27	22
R5	Jun-08	8.5	21	23
R6	Dec-08	9.2	24	10
R7	Jun-09	8.0	56	55
R8	Dec-09	7.8	65	20
R9	Jun-10	3.0	81	53
R10	Dec-10	8.1	39	16
R11	Jul-11	4.5	94	56
R12	Dec-11	11	25	12
R13	Jun-12	6.9	170	66
R14	Dec-12	12	68	21
R15	Jul-13	14	38	7.9
R16	Dec-13	8.4	64	17
R17	Jun-14	3.4	130	55
R18	Dec-14	7.4	120	38
R19	Jun-15	9.0 J	57	15
R20	Dec-15	13	27	ND
R21	Jun-16	5.4	61	25

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	490	35	0.51 J
Q2	Mar-07	440	42	9.7
Q3	Jun-07	410	61	18
Q4	Nov-07	510	90	24
R5	Jun-08	440	90	12
R6	Dec-08	410	79	13
R7	Jun-09	330	68	9.3
R8	Dec-09	350	140	21
R9	Jun-10	330	170	15
R10	Dec-10	310	120	15
R11	Jul-11	0.52 J	12	2.6
R12	Dec-11	2.3	56	4.3
R13	Jun-12	280	140	11
R14	Dec-12	280	100	5.9
R15	Jul-13	300	110	2.6
R16	Dec-13	370	140	9.6
R17	Jun-14	190	110	9.6
R18	Dec-14	260	150	16
R19	Jun-15	200 J	100	6.1
R20	Dec-15	170	150	11
R21	Jun-16	170	95	5.5

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	4.0	54	0.6 J
Q2	Mar-07	2.8	38	ND
Q3	Jun-07	3.1	60	2.6
Q4	Nov-07	3.8	39	ND
R5	Jun-08	2.4	48	1.9
R6	Dec-08	2.2	34	0.26 J
R7	Jun-09	1.7	32	2
R8	Dec-09	1.7	28	1.6
R9	Jun-10	0.39 J	33	3.8
R10	Dec-10	0.53 J	30	7.7
R11	Jul-11	0.38 J	37	7.9
R12	Dec-11	0.82 J	21	2.9
R13	Jun-12	0.87 J	30	2.8
R14	Dec-12	1.1	18	ND
R15	Jul-13	1.6	24	0.83 J
R16	Dec-13	1.3	23	1.8
R17	Jun-14	1.3	23	1.7
R18	Dec-14	0.85 J	13	ND
R19	Jun-15	0.48 J	18	2.1
R20	Dec-15	0.74 J	18	1.2
R21	Jun-16	ND	18	3.6

Round	Date	TCE ug/L	cis-DCE ug/L	VC ug/L
Q1	Jan-07	ND	1.2	ND
Q3	Jun-07	ND	1.7	ND
R5	Jun-08	ND	1.3	ND
R6	Dec-08	0.33 J	0.4 J	ND
R7	Jun-09	ND	1.0	ND
R8	Dec-09	ND	0.56 J	ND
R9	Jun-10	ND	0.61 J	ND
R10	Dec-10	ND	0.86 J	ND
R11	Oct-11	ND	2.3	ND
R12	Dec-11	ND	0.95 J	ND
R13	Jun-12	ND	2.2	ND
R14	Dec-12	ND	0.85 J	ND
R15	Jul-13	ND	2.2	ND
R16	Dec-13	ND	1.7	ND
R17	Jun-14	ND	0.98 J	ND
R18	Dec-14	ND	0.89 J	ND
R19	Jun-15	ND	1.1	ND
R20	Dec-15	ND	1.4	ND
R21	Jun-16	ND	2.8	ND

NOTE:

ND = NON-DETECT

LEGEND:

- PAVED ROAD
- DIRT ROAD
- GROUND CONTOUR AND ELEVATION (650)
- TREE
- WETLAND & DESIGNATION (W-B)
- MONITORING WELL AND DESIGNATION (PT-22)
- RAILROAD TRACKS
- BRUSH
- CHAIN LINK FENCE
- UTILITY POLE
- APPROXIMATE LOCATION OF FIRE HYDRANT
- FUEL OR UNDERGROUND STORAGE TANK
- SURVEY MONUMENT (SEAD-1 EL.630.90')
- APPROXIMATE LOCATION OF WATER MAIN
- PILOT STUDY BIOWALL (2005)
- SINGLE BIOWALL (2006)
- DOUBLE-WIDE BIOWALL (2006)
- ZERO VALENT IRON WALL (1998)
- GROUNDWATER ISOCONTOUR (UG/L) BASED ON JANUARY 2000 DATA (-10-)
- OFF-SITE PERFORMANCE MONITORING WELL IN L.T.M. PROGRAM (MW-56)
- ON-SITE PLUME PERFORMANCE MONITORING WELL IN L.T.M. PROGRAM (PT-22)
- BIOWALL PROCESS MONITORING WELL IN L.T.M. PROGRAM (MWT-26)

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT

ASH LANDFILL

LONG TERM MONITORING REPORT

DEPT. ENVIRONMENTAL ENGINEERING | Dwg. No.

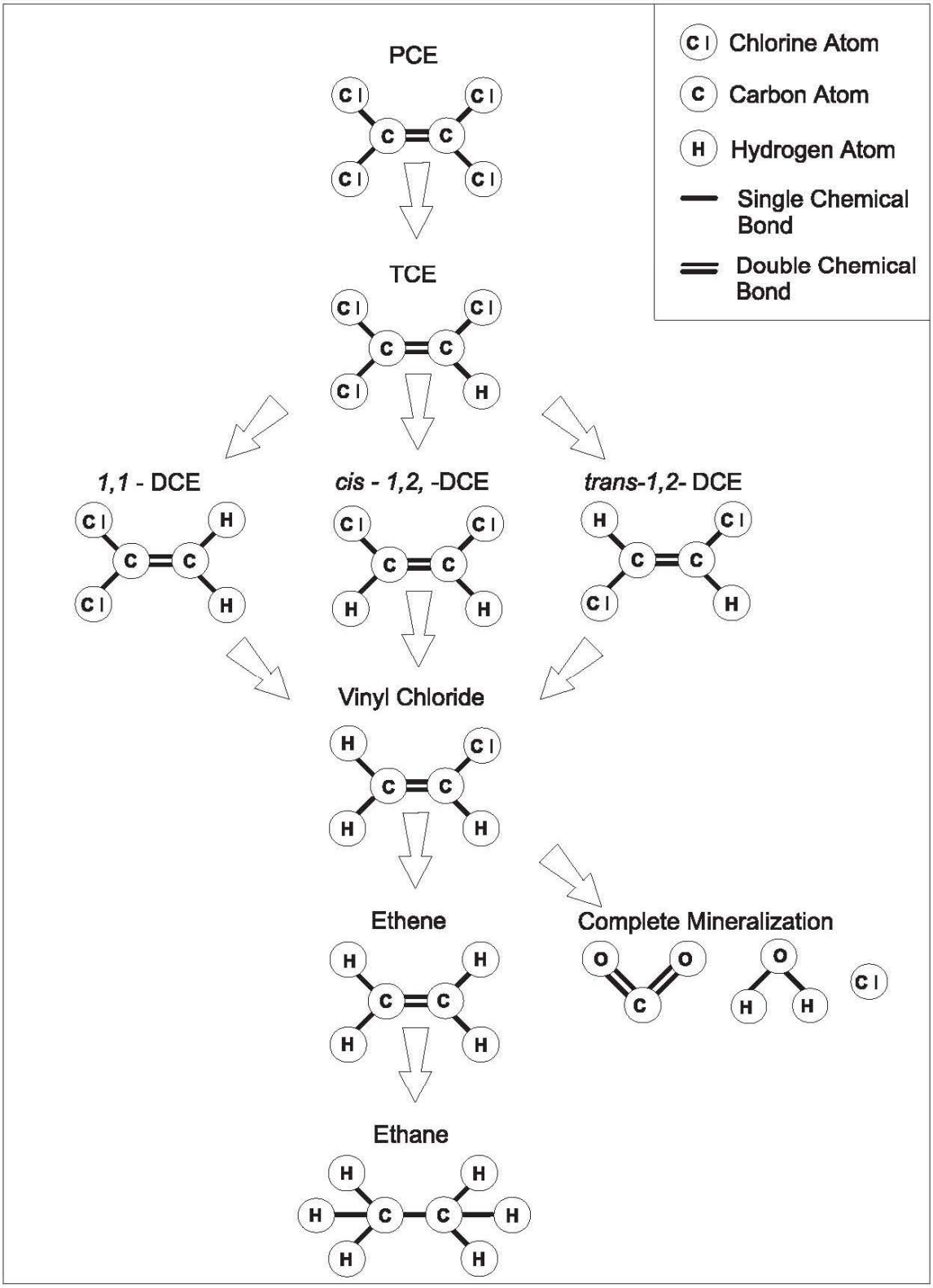
FIGURE 2

CHLORINATED ETHENES CONCENTRATIONS IN GROUNDWATER

SCALE | DATE: JUNE 2016 | REV: -

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\ASH LANDFILL LTM\ROUND 21 - JUNE 2016\R21 LETTER REPORT\DRAFT\, DATE: 07/07/2016 12:56:15PM, P0091241

Figure 3
Reductive Dechlorination of Chlorinated Ethenes
Ash Landfill Annual Report
Seneca Army Depot Activity



(USEPA, 1998)

LEGEND:

- PAVED ROAD
- DIRT ROAD
- GROUND CONTOUR AND ELEVATION
- TREE
- WETLAND & DESIGNATION
- MONITORING WELL AND DESIGNATION
- RAILROAD TRACKS
- BRUSH
- CHAIN LINK FENCE
- UTILITY POLE
- APPROXIMATE LOCATION OF FIRE HYDRANT
- FUEL OR UNDERGROUND STORAGE TANK
- SURVEY MONUMENT
- ABANDONED MONITORING WELL
- APPROXIMATE LOCATION OF WATER MAIN
- PILOT STUDY BIOWALL (2005)
- SINGLE BIOWALL (2006)
- DOUBLE-WIDE BIOWALL (2006)
- ZERO VALENT IRON WALL (1998)
- GROUNDWATER CONTOUR
- GROUNDWATER FLOW DIRECTION

70 0 70 140
(FEET)

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT
ASH LANDFILL
LONG TERM MONITORING REPORT

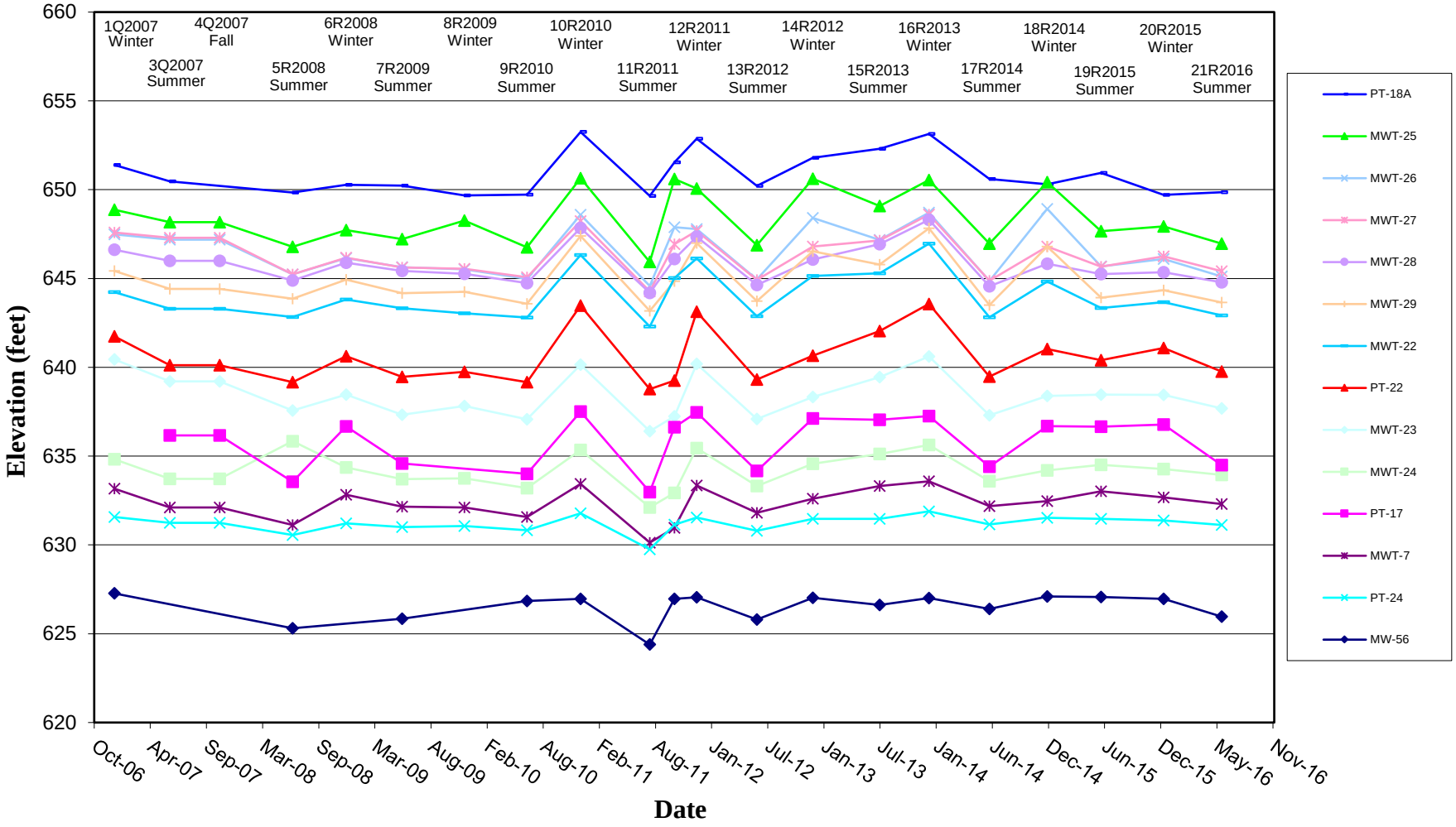
DEPT.	Dwg. No.
ENVIRONMENTAL ENGINEERING	

FIGURE 4
ASH LANDFILL GROUNDWATER CONTOURS & GROUNDWATER FLOW DIRECTION JUNE 2016

SCALE	DATE	REV
	AUGUST 2016	–

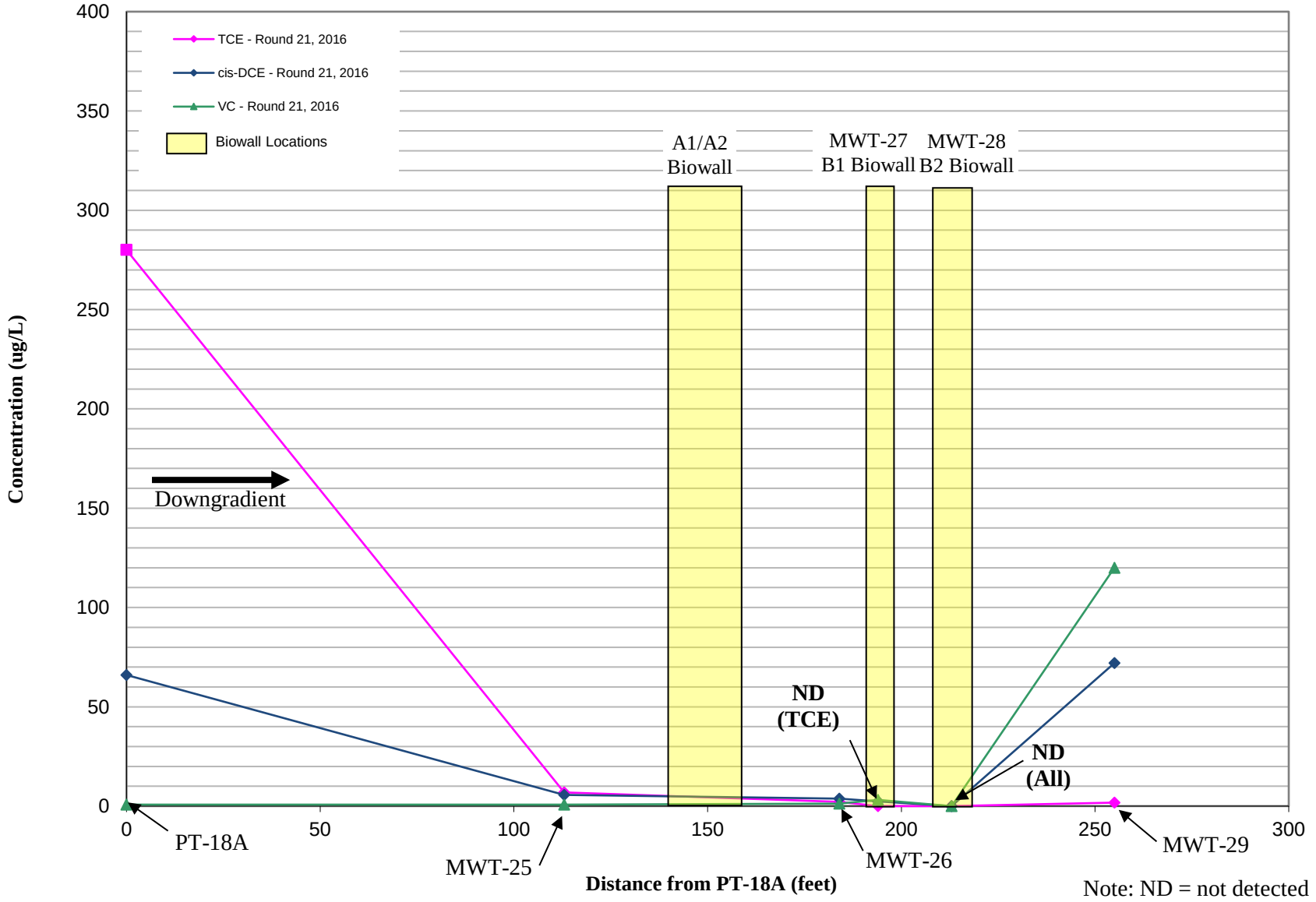
FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\ASH LANDFILL LTM\ROUND 21 - JUNE 2016\R21 LETTER REPORT\DRAFT\FIGURES\FIGURE 4.DWG, DATE: 07/19/2016 09:50:06AM, p0018397

Figure 5
Groundwater Elevations Rounds 1 through 21
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity



Notes: Groundwater levels were measured on: Dec 12-15, 2006; Jun 4, 2007; Nov 7, 2007; Jun 23, 2008; Dec 23, 2008; Jun 1, 2009; Dec 14, 2009; Jun 28, 2010; Dec 13, 2010; Dec 12, 2011; Jun 18, 2012; Dec 10, 2012; Jul 8, 2013; Dec 9,2013; Jun 17, 2014; Dec 15, 2014; Jun 2,2015, Dec 15, 2015, and Jun 14, 2016.
In Round 11, Groundwater levels were collected on July 18, 2011, and again on Oct 3, 2011 when Parsons returned to sample MW-56. Groundwater elevations were not measured at well MW-56 during 3Q2007, 4Q2007, 6R2008, or 8R2009; at PT-17 during 1Q2007 or 8R2008; or at PT-18A during 4Q2007. Groundwater levels were not recorded during 2Q2007.

Figure 6
Concentrations of VOCs Along the Biowalls
Round 21 - June 2016
Ash Landfill Long-Term Monitoring
Seneca Army Depot Activity



Note: ND = not detected

DRAFT

2015 LONG-TERM MONITORING ANNUAL REPORT

FOR THE OPEN BURNING GROUNDS
SENECA ARMY DEPOT ACTIVITY, ROMULUS, NEW YORK

Prepared for:

U.S. ARMY, CORPS OF ENGINEERS, ENGINEERING AND SUPPORT CENTER, HUNTSVILLE, ALABAMA

U.S. ARMY, CORPS OF ENGINEERS, NEW YORK DISTRICT NEW YORK, NEW YORK

and

SENECA ARMY DEPOT ACTIVITY ROMULUS, NEW YORK

Prepared by:

PARSONS
100 High Street
Boston, MA 02110

Contract Number W912DY-08-D-0003
Task Order No. 0015
EPA Site ID# NY0213820830
NY Site ID# 8-50-006

March 2016

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1.0 INTRODUCTION

This Annual Report has been prepared by Parsons Government Services, Inc. (Parsons) on behalf of the United States Army Corps of Engineers, Engineering and Support Center – Huntsville (USAESCH) and the Seneca Army Depot Activity (SEDA or the Depot) to provide a review of the long-term monitoring (LTM) activities conducted in October 2015 for the Open Burning (OB) Grounds (the Site) located at SEDA in Seneca County, New York; and to provide recommendations for future LTM at the Site.

The Record of Decision (ROD) for the OB Grounds was signed in 1999, and presented the selected remedy for addressing potential exposure to elevated levels of metals (specifically lead and copper) in the Site soils and the sediments of the adjacent Reeder Creek (Parsons, 1999). The remedy specified in the ROD is described in **Section 2.3** of this report.

Presently, quantitative monitoring of sediment quality (i.e., submitting samples for copper and lead analysis as identified in the approved remedy for the Site in the ROD) is not included as part of the LTM activities, and is discussed in further detail in **Section 1.1** of this report. In accordance with the approved remedy as presented in the ROD, the current LTM activities at the Site include the following three components:

- The annual collection and analysis of groundwater samples for lead and copper concentrations;
- The inspection of the vegetated, compacted soil cover that has been constructed over interred lead-contaminated soil as part of the Site remedial actions in order to assess if erosion or breaching of the protective cover has occurred, which could result in the potential migration of contaminated soil; and
- The inspection of Reeder Creek where the Creek abuts the OB Grounds to evaluate the potential for inward migration and deposition of soil from the OB Grounds.

This report presents and summarizes the results of the most recent annual LTM event performed in October 2015 and provides recommendations for future LTM at the OB Grounds.

1.1 Long-Term Monitoring Activities

The OB Grounds LTM activities are being performed in accordance with the *Long-Term Monitoring Plan for the Open Burning Grounds, Final* (LTM Plan) (Parsons, 2007). Long-term monitoring activities include the collection of groundwater quality data to monitor the effectiveness of the implemented remedy at the Site for preventing future impacts to groundwater at the OB Grounds and to sediments in Reeder Creek. Additionally, monitoring of the vegetated compacted soil cover placed over the contaminated soils at the OB Grounds is required to assure the long-term integrity of the soil cover, including the potential mobilization and migration of lead-contaminated soil buried beneath the cover; and to prevent direct contact with, and incidental ingestion of, soils containing lead at concentrations up to 500 mg/kg by terrestrial wildlife at the Site.

Part of the OB Grounds LTM program includes a qualitative assessment (i.e., visual inspection) of Reeder Creek for evidence of migration of material via surface water flow or groundwater transport of contaminants into the remediated section of Reeder Creek adjacent to and down gradient of the OB

Grounds. The visual inspection consists of walking the creek bed (or embankment) to look for evidence of soil erosion or sloughing from the Creek embankment adjacent to the OB Grounds and/or the accumulation of sediment along the stream bed. Additionally, groundwater transport of contaminants is monitored by the annual groundwater sampling of the OB Grounds wells. Presently, quantitative monitoring of sediment quality (i.e., submitting samples for copper and lead analysis as identified in the approved remedy for the Site in the ROD) is not included as part of the LTM activities; the U.S. Army Corps of Engineers (Army), the U.S. Environmental Protection Agency (EPA), and the New York State Department of Environmental Conservation (NYSDEC) agreed that until data indicated that either groundwater transport of contaminants or soil transport from the OB Grounds was occurring, sampling and analysis of Creek sediments would not be required.

Long-term monitoring began at the OB Grounds site in November 2007 (**Exhibit 1.1**). LTM at the OB Grounds site was initially scheduled to occur on a quarterly basis. The results of the first four LTM rounds were combined and summarized in an annual report, in which, the recommended frequency of monitoring was recommended to change from quarterly to annually. Based on comments received from EPA and NYSDEC in 2009, the Army authorized the performance of an inspection of Reeder Creek. The monitoring frequency of groundwater was agreed upon by EPA and NYSDEC in February 2010 to be conducted annually. Subsequent to Round 5, investigations at the OB Grounds have included yearly groundwater sampling and inspection of both the soil caps and Reeder Creek.

Exhibit 1.1 – LTM and Inspection Summary

Round Number	Event	Date	Report Title
1	LTM	November 21-28, 2007	Final, OB Grounds Long-Term Monitoring Annual report and One Year Review (Parsons, 2009).
	Cover Inspections	January 11, 2008	
2	LTM and Cover Inspections	February 25-26, 2008	
3	LTM and Cover Inspections	May 20-21, 2008	
4	LTM and Cover Inspections	August 25-26, 2008	
5	LTM and Cover Inspections	August 2-3, 2010	Draft Final, 2010 Long-Term Monitoring Annual Report for the Open Burning Grounds, Seneca Army Depot Activity (Parsons, 2011).
6	LTM, Cover Inspections, and Inspection of Reeder Creek	October 3-6, 2011	Final, 2011 Long-Term Monitoring Annual Report for the Open Burning Grounds, Seneca Army Depot Activity (Parsons, 2013).
7	LTM, Cover Inspections, and Inspection of Reeder Creek	October 8-10, 2012	Final, 2012 Long-Term Monitoring Annual Report for the Open Burning Grounds, Seneca Army Depot Activity (Parsons 2014a).
8	LTM, Cover Inspections, and Inspection of Reeder Creek	December 9-14, 2013	Draft, 2013 Long-Term Monitoring Annual Report for the Open Burning Grounds, Seneca Army Depot Activity (Parsons 2014b).
9	LTM, Cover Inspections, and Inspection of Reeder Creek	October 14-16, 2014	Final, 2014 Long-Term Monitoring Annual Report for the Open Burning Grounds, Seneca Army Depot Activity (Parsons, 2015).
10	LTM, Cover Inspections, and Inspection of Reeder Creek	October 19-21, 2015	Draft, 2015 Long-Term Monitoring Annual Report for the Open Burning Grounds, Seneca Army Depot Activity

2.0 SITE BACKGROUND

2.1 Site Description

The Depot is a 10,587-acre former military facility located in Seneca County in the towns of Varick and Romulus, New York, and was owned by the United States Government and operated by the Department of the Army between 1941 and 2000 (**Figure 1**). In 2000, the Army closed the Depot and assumed a caretakers' role of the property, pending the closeout of its continuing environmental obligations and the leasing or transfer of property to other public or private parties for beneficial reuse purposes. Since 2000, more than 8,250 acres of land have been transferred to other parties.

The Depot is located between Seneca Lake and Cayuga Lake and is bordered by sparsely populated farmland and New York State Highway 96 to the east, by New York State Highway 96A to the west, and by sparsely populated farmland to the north and south. The OB Grounds is located in the northwestern portion of the Depot where the planned future use of the land currently is designated for conservation purposes **(Figure 1)**. As situated, the OB Grounds sits a minimum of 1,780 feet away from the nearest Depot boundary, which is located to the west of the area of concern (AOC). The OB Grounds site sits on gently sloping terrain and is bounded on the east by Reeder Creek, a perennial creek that is generally less than 1 foot deep and which eventually flows into Seneca Lake (**Figure 2**). The quality of surface water in Reeder Creek is designated by the State of New York as a Class C water body (best usage of fresh water is fishing; the waters shall be suitable for fish propagation and survival). Seneca Lake is located approximately 10,000 feet west of the OB Grounds site and is used as a source of drinking water for numerous surrounding communities and the Depot.

The OB Grounds is vegetated with grass and brush and there are no permanent structures within the area other than small concrete bunkers and a metal garage structure. The former Open Detonation Area (SEAD-45) is located immediately north of the OB Grounds, and the former Explosive Ordnance Disposal Area (SEAD-57) is located approximately 4,000 to 5,000 feet south of the former OB Grounds. A Site plan of the former OB Grounds prior to the removal of contaminated soil is provided as **Figure 2**. The OB Grounds was historically used for surface burning of explosive trash and propellants.

2.2 Site Geology and Hydrology

The stratigraphy of the OB Grounds generally consists of between 2 and 10 feet of glacial till underlain by a zone of weathered bedrock (shale). The depth to groundwater in the till/weathered shale aquifer varies seasonally between approximately 2 and 7 feet below the ground surface (bgs). Infiltration of precipitation is the sole source of groundwater for the overburden aquifer. The direction of the groundwater flow in the till/weathered shale aquifer at the OB Grounds is generally to the east towards Reeder Creek (**Figure 3)**.

Historic groundwater elevation monitoring in wells located at the OB Grounds prior to the remedial action indicated the presence of a groundwater divide near the western edge of the Site. The approximate location of the apparent groundwater divide found in April 1993 is highlighted on **Figure 3** and represents a high point of the upgradient groundwater flow regime. The divide diverts a portion of the groundwater to the west, or away from Reeder Creek, which lies to the east of the divide. Historic sampling results

from wells located west of the identified divide suggest that the quality of groundwater has not been impacted by soils at the OB Grounds.

Pre-remedial action surface water drainage from the OB Grounds was primarily to the east-northeast via a series of man-made drainage ditches, culverts, and spillways to Reeder Creek. During the remedial action, many of the drainage ditches and culverts were destroyed or filled, altering the surface flow patterns. Additionally, the historic surface water spillways connecting the OB Grounds and Reeder Creek were modified during the remedial action to include ditch breaks to prevent soil transport to the creek.

Presently, little, if any, storm event runoff impacting the former OB Grounds reaches the creek via overland flow because it is captured in one of the numerous, localized topographic lows that are scattered throughout the AOC. The topographic lows result from the remedial action performed at the AOC described in **Section 2.3**. The captured storm water subsequently infiltrates into the soil or evaporates.

2.3 Summary of the Remedial Action

The remedy specified in the ROD for the OB Grounds included:

- Removal of the berms surrounding the historic burn pads;
- Removal of all soils to a depth of at least 1 foot;
- Placement of a 9-inch vegetative cover over any soils with lead concentrations greater than 60 mg/kg, but less than or equal to 500 mg/kg;
- Excavation of sediments in Reeder Creek with elevated levels of copper or lead; and
- Implementation of a monitoring program for groundwater, sediment, and the capped areas.

The first four of these required remedial actions were conducted between June 1999 and May 2004 by Weston Solutions Inc. Currently, the LTM component of the remedy is being implemented by Parsons. Long-term groundwater monitoring at the Site commenced in November 2007 followed by inspections of the cover commencing in January 2008 (**Exhibit 1.1**).

The overall objective of the OB Grounds LTM program is to monitor the effectiveness of the remedial action completed at the Site with respect to preventing future groundwater quality deterioration and the erosion or breaching of the vegetated soil cover. The purpose of the soil cover is to (1) prevent incidental contact and ingestion of contaminated soil left in place at the Site, and (2) prevent the potential mobilization and migration of lead-contaminated soil interred beneath the cover. In addition to assessing the quality of Site groundwater and the integrity of the cover, the results of the periodic monitoring will be used to assess the need for the design and implementation of any sediment monitoring program that may subsequently be needed to assess potential Site impacts to the sediment quality found in Reeder Creek per the requirements set forth in the ROD.

3.0 LONG-TERM GROUNDWATER MONITORING

Long-term groundwater monitoring at the OB Grounds began in November 2007. The initial monitoring frequency was quarterly, but subsequent to EPA and NYSDEC approval, the frequency was changed to annual sampling beginning in Round 5. Monitoring rounds, their dates and associated annual reports are summarized in **Exhibit 1.1** The most recent LTM event, Round 10, was performed from October 19 to 21, 2015. Six monitoring wells (MW23-1, MW23-2, MW23-3, MW23-4, MW23-5, and MW23-6), which were installed in 2007 to replace the historic monitoring well network that existed at the Site prior to the remedial action, were gauged and sampled as part of this monitoring event. The results of this most recent round (Round 10) are presented in this Report.

For each sampling round conducted at the OB Grounds, groundwater samples were collected using low-flow sampling techniques. Sampling procedures, sample handling and custody, holding times, and collection of field parameters were conducted in accordance with the *Final Sampling and Analysis Plan for Seneca Army Depot Activity* (SAP) as well as the Quality Assurance Project Plan (QAPP) which is included within the SAP (Parsons, 2005). The selected laboratory has the capability to conform to the project QAPP and has a current DoD Environmental Laboratory Accreditation Program (DoD ELAP) certification in which the laboratory demonstrated its competency and document conformance to the current DoD Quality Systems Manual for Environmental Laboratories (DoD QSM).

During each monitoring round, groundwater samples and groundwater elevation measurements were collected from the six wells located at the OB Grounds. Groundwater samples for Round 10 were collected and submitted to TestAmerica in Savannah, Georgia for the analysis of total copper and total lead by USEPA SW846 Method 6010C. Analytical results reported for total copper and total lead were compared to Site-specific action levels provided in **Table 1**.

Groundwater quality parameters listed below were measured and recorded prior to sample collection and the groundwater samples were collected once parameters had stabilized within 10 percent:

- pH
- Oxidation/reduction Potential (ORP)
- Dissolved oxygen (DO)
- Conductivity
- Temperature
- Turbidity

The pH, ORP, conductivity, and temperature of the groundwater were measured with a Horiba U-52 water quality meter; turbidity was measured with a Hach 2020 Turbidity Meter; and DO content was measured with an YSI 85 Dissolved Oxygen Meter. Field parameters were measured approximately every five minutes in order to assess when the well was adequately purged and the groundwater conditions had stabilized prior to sample collection, and to assess macro-groundwater quality.

3.1 Groundwater Elevations

Groundwater levels were recorded during each LTM round and elevation data from each event are presented in **Table 2**. Groundwater levels were measured prior to the collection of groundwater samples. Field forms of the groundwater elevations measured from the most recent event are provided in **Appendix A**.

The present groundwater flow patterns across the Site are interpreted via evaluation of the October 2015 (Round 10) groundwater elevation data and the historic pre-remedial action groundwater data. Groundwater elevation data collected from Round 10 confirm a general east to northeast groundwater flow direction across the Site as illustrated by historic groundwater contours developed from groundwater elevation data collected in April 1993 (**Figure 3**). The elevations observed in the western portion of the site (wells MW23-4 and MW23-5) continue to be higher than those recorded in the eastern portion (MW23-1 through MW23-3) (**Table 2**). Along the eastern boundary of the OB Grounds, in proximity to Reeder Creek, the groundwater elevations measured at MW23-2 in the center of the boundary continue to appear higher than those measured at MW23-1 (located to the southeast of MW23-2 along the boundary) and MW23-3 (located to the northwest of MW23-2 along the boundary) (**Figure 3**). The data suggest flow variations to the south and the north along the Site/Reeder Creek boundary. The October 2015 groundwater elevations were observed to be within the historic range of maximum and minimum groundwater elevations from the site (**Figure 4**).

3.2 Analytical Data

The analytical results from the groundwater samples collected during Round 10 are presented in **Table 3** and are compared to the groundwater cleanup goals listed in **Table 1**. **Appendix B** presents the analytical results from each round of LTM. The laboratory data sheets for Round 10 are provided in **Appendix C**. The data validation for the October 2015 (Round 10) sampling can be found in **Appendix D** of this report. Round 10 groundwater samples were validated according to USEPA Region 2's *ICP-MS Data Validation for Contract Laboratory Program based on SOW ILMO5.3, HW-2b Revision 15* (USEPA, 2012). The data validation did not report any non-compliance issues in the data package.

In the samples collected during Round 10, total lead was not detected above the applicable EPA maximum contaminant limit (MCL) action level of 15 µg/L for groundwater. Total copper was not detected above the applicable NYSDEC Class GA Groundwater Standard of 200 µg/L in the samples collected during Round 10. Total copper and total lead were not detected above their reporting limit (RL) in Round 10 (**Table 3**). **Figures 5** through **10** present a summary of the groundwater sampling results for monitoring wells MW23-1 through MW23-6 from each round of monitoring conducted following the completion of the remedial action.

The LTM data supports that groundwater at the Site has not been impacted by residual levels of copper and lead that remain in the soils at the Site. Total copper has not been detected above its RL in the groundwater during any of the post remedial action sampling rounds. Total lead has not been detected in the groundwater above the action level of 15 µg/L during any of the post remedial action sampling rounds. Six of the seven lead detections have been estimated concentrations and the maximum concentration of lead detected in ten rounds of sampling was 5.4 µg/L at well MW23-4 during Round 2 (**Appendix B**). Evaluation of the water quality parameters measured at Site wells during current (and previous) LTM activities indicate generally mild alkaline conditions, which suggest that lead should not be readily mobile in groundwater under current Site conditions.

3.3 Statistical Analysis

Subsequent to the removal action, ten rounds of LTM sampling have been completed at the OB Grounds since the end of 2007. During this time, 70 groundwater samples (including duplicates) were analyzed for total copper and total lead (**Appendix B**). Total copper was not detected above its RL in any of the samples collected. Total lead was detected in 7 of 70 samples (including two duplicates); none of the concentrations exceeded the EPA MCL action level and 6 of 7 detections were estimated concentrations. To quantify the LTM results over time and examine any trends in the data, a statistical analysis was performed on the data using EPA ProUCL version 5.0 software (USEPA, 2015). ProUCL results are provided in **Appendix F**. No statistical tests were performed on the total copper results as there were no detects.

Limited detections of total lead have been observed at wells MW23-4, MW23-5, and MW23-6 during three of the ten sampling rounds (i.e. Rounds 2, 5, and 6). A summary of the detections during the ten sampling rounds is provided in **Exhibit 3.1**. A comprehensive list of all groundwater sample results (total copper and total lead) from the ten rounds of LTM at the OB Grounds is available in **Appendix B**.

Exhibit 3.1 – Summary of Total Lead Detections in Groundwater

Round, Date	Well ID	Result [µg/L]
LTM Round 2, February 2008	MW23-4	5.4
LTM Round 5, August 2010	MW23-4	2.7 J
	MW23-5	2.4 J *
	MW23-6	3.6 J
LTM Round 6, October 2011	MW23-5	1.1 J
	MW23-6	1.2 J
	MW23-6	1.5 J *

Note: Results marked with an asterisk were obtained from duplicate samples.

At this time, the number of points in the dataset for each well does not meet the minimum data requirements (4 to 9 detected data points for limited tests; 10 data points for all tests) for any meaningful or reliable conclusions. The limited number of detected concentrations makes a statistical analysis not possible and supports the argument that total lead has not been migrating from the soil matrix to the groundwater. After ten annual rounds of sampling, the migration of COCs from the soil into the groundwater has not been observed.

4.0 SOIL COVER INSPECTION

The cover inspection consisted of documenting observations of the twenty-five (25) 125-foot by 125-foot grids, where soils with residual lead concentrations between 60 mg/kg and 500 mg/kg were interred under a 9 inch-thick soil cover. The locations of the grids are shown on **Figure 11**, which is based on a figure provided by Weston Solutions in the "Completion Report for the Open Burning Grounds Soil and Sediment Remediation" (Weston Solutions, 2005) and a 2011 aerial image of the OB Grounds obtained from Bing.com. The October 2015 cover inspection log and the previous year's (October 2014) inspection log are presented in **Table 4**. Inspection forms documenting the Round 10 soil cover inspection at the Site are provided in **Appendix A**. Photographs from the cap inspections are included in **Appendix G**. Observations made during the cover inspection completed on October 19, 2015 are provided below.

4.1 October 2015

The OB Grounds soil covers were inspected on October 19, 2015 (**Appendix A**). No animal burrowing activity was observed in any of the capped areas. Signs of past minor erosion, as noted in the 2014 Annual Report (October 2014 Inspection), continue to be observed along the sloped edges of Grid I8 adjacent to the drainage ditch (between Grids J8 and J9) as a result of surface water run-off from the western portion of the Site towards Reeder Creek. However, the erosion area has not grown in size or depth. The sloped edges of Grid I8 were also observed to have lower vegetation density than the rest of the Grid. Overall, the erosion along the edges of the soil cover in Grid I8 has not changed since the October 2014 inspection and no corrective action is warranted at this time. The condition of this location will be reassessed during the next inspection event to determine if corrective measures are needed.

Signs of minor erosion were observed where the soil cover transitions to the native ground surface at the western edge of the soil cover within Grid I7 and at the northern edge of the soil cover within Grid I6 (**Table 4**). These areas where signs of minor erosion had been observed had lower vegetation density than the rest of the respective Grids. The condition of these locations will be reassessed during the next inspection event and no corrective action is warranted at this time.

The northeast corner of Grid A5 and east side of Grid D7 contained areas with sporadic vegetation (**Table 4**). Each of these grids had areas which were not as densely vegetated as the surrounding area. In each case, no disturbances to the soil cap were observed, and no signs of erosion were evident. The condition of these locations were similar to conditions observed in October 2014 and previous inspections. The condition in these areas will be reassessed during the next inspection event. No corrective action is warranted at this time.

The shallow tire ruts in Grid C7 which had been regraded and filled with crushed shale following the October 2014 inspection were in good condition (**Appendix G, Photo 8**). No disturbances to this corrective measure or the remaining sections of the soil cap in Grid C7 were observed. The condition of the corrective measure will be reassessed during the next inspection event.

5.0 REEDER CREEK INSPECTION

Accessible portions of Reeder Creek adjacent to the OB Grounds were inspected by walking along the streambed the length of the inspection area and making observations of the creek's condition. A section of the Reeder Creek embankment which was previously cleared of vegetation as part of the 2012 OD Grounds Munitions Response Action project continues to show new vegetative growth as observed since the 2013 inspection. Observations made during the October 19, 2015 inspection are provided below.

5.1 October 2015

A visual inspection of the Reeder Creek streambed was conducted on October 19, 2015 at locations adjacent, down-gradient, and up-gradient to the OB Grounds. Per the requirements set forth in the Site-Specific Health and Safety Plan, personal protective equipment and any additional health and safety equipment was used as appropriate. Photos of Reeder Creek were taken to document the current condition of the creek and its embankments. Photo locations are shown on **Figure 12** and Photos 1 through 37 are provided in **Appendix E**.

Overall, the conditions of Reeder Creek at locations down-gradient and adjacent to the OB Grounds were observed to consist of the exposed bedrock streambed and miscellaneous fracture shale pieces with sections containing sediment and other sections covered with thin, brown slime-like material similar to what was observed during previous annual inspections. Based on field observations, the source of the sediment is believed to be from decomposition of leaves and other organics that have accumulated within the creek bed. Based on several observations during the Reeder Creek inspection, large rain events and high water levels have contributed to the erosion at the base of the Reeder creek embankments in some areas **(Appendix E – Photos 7, 15, 27, 30, and 33).** Some portions of the Reeder Creek streambed from the OD Grounds to up-gradient of OB Grounds were not accessible due to high water levels.

The inspection started at the down-gradient section of Reeder Creek within the adjacent OD Grounds and proceeded upstream. The embankments and creek bottom were inspected as the inspection team progressed upstream (**Figure 12**). Sediment was observed down-gradient of the OB Grounds in areas that were outside the prior creek bed excavation areas. A thin brown slime-like material, measuring only a few millimeters thick, was observed on the creek bottom (similar to the previous inspections). The beaver dam first observed in the December 2013 inspection and seen again in October 2014 was no longer standing (**Appendix E – Photo 13**). The majority of the branches making up the dam are no longer present and were likely swept away from an apparent large scale rainfall event. During the time of the inspection, the depth of water within the creek was approximately 3-4 inches deep, but in certain areas the water was up to 2 feet deep (**Appendix E – Photo 22**).

No evidence was observed that showed materials from the sidewalls of the Reeder Creek embankments had collapsed into the creek. The embankments were very well vegetated, aiding in the prevention of any sidewall collapse and sediment transport. However, local erosion was apparent at the base of the embankments in several areas and was likely the cause of elevated water levels and accelerated currents during strong rain fall events.

Examination of the spillways, where surface water from the OB Grounds discharges to Reeder Creek, found no visible evidence that overland surface water flow had transported soils from the OB Grounds

into Reeder Creek. However, large rainfall events during the previous year lead to small flow pathways forming around the erosion control sandbags aligned in the spillway ditch located between MW23-1 and MW23-2 (**Appendix E – Photo 37**). The spillways were free of accumulation of excessive soil, but debris in the form of tree branches were observed near the culvert leading down into Reeder Creek. Field observations noted that the mechanisms previously placed at the OB Grounds to prevent transported soil material from entering the spillways were still working as intended. However, the Army was informed during the LTM event that the erosion control sandbags should be reinforced and/or realigned to account for potential future large precipitation events.

5.2 Inspection Observations

As reported above, the groundwater data collected during historic sampling events as well as during the tenth round of the Long-Term Monitoring Program shows no evidence of a release of total copper or total lead from the OB Grounds Site. Previous soil cover inspections did reveal that occasional animal burrows and shallow erosion depressions were present in the cover at the contaminated soil burial areas, but none of the past noted burrow holes or depressions were sufficiently sized to allow buried soils to escape their containment (these noted holes and depressions were repaired in August 2008 as part of the Army's continuing maintenance activities). Based on the October 2015 inspection, there were no visible signs that OB Grounds site soils are being released via overland flow to Reeder Creek. As such, the Army does not see any evidence to suggest that a release of lead or copper above background levels is occurring from the OB Grounds site. The past detections of lead (below the action level) were located on the western edge of the OB Grounds (MW23-4 and MW23-5) and south of the OB Grounds (MW23-6). The absence of detectable concentrations of lead and copper in the three wells (MW23-1, MW23-2, and MW23-3) immediately adjacent to Reeder Creek supports the observation that Reeder Creek has not been impacted by lead or copper.

Based on these data and this information, the Army has not conducted sediment sampling and analysis of Reeder Creek as part of the LTM at the OB Grounds. The Army will conduct another visual inspection of the creek bed and spillways connecting the OB Grounds to Reeder Creek during the next scheduled annual monitoring event. If evidence of overland transport of soil or groundwater migration of contaminants from the OB Grounds to Reeder Creek is identified, a plan will be prepared and submitted for approval which will identify a sediment monitoring program.

6.0 LONG-TERM MONITORING CONCLUSIONS AND RECOMMENDATIONS

The following conclusions can be made based on the results of the October 2015 (Round 10) of LTM at the OB Grounds:

- Residual lead and copper concentrations remaining in the soils have not impacted groundwater at, or in the immediate vicinity of the Site above the applicable action levels.
- During ten rounds of groundwater sampling, copper and lead concentrations have not been detected above their RL enough times to perform a meaningful statistical analysis of the historical data thus indicating little to no migration of these COCs into the groundwater.
- The integrity of the vegetated soil cover overlying interred contaminated soils at the OB Grounds Site was intact and there was no evidence that terrestrial wildlife are exposed or will be exposed to the lead-contaminated soils interred below the 9-inch soil cover.
- The Army will continue to monitor soil cover erosion and will note any instance of cover erosion or exposed native or interred soil.
- Based on evaluation of the groundwater data and the results of the cover inspection, there is no evidence to suggest that the OB Grounds may be contributing to the degradation of sediment quality in Reeder Creek.
- Field observations noted that the mechanisms previously placed at the OB Grounds to prevent transported soil material from entering the spillways were still working as intended.
- The Army will continue to inspect Reeder Creek for evidence of sediment deposition and if it is observed, a sediment sampling and analysis program plan will be prepared, submitted for approval, and implemented for Reeder Creek at locations adjacent to the OB Grounds.

Based on the results of the LTM sampling events conducted at the OB Grounds, the Army recommends discontinuing LTM of the groundwater. The recommendation to terminate LTM groundwater sampling is included as part of the Five Year Review Report for the Former Solid Waste Management Units at Seneca Army Depot Activity which will be submitted under separate cover in 2016. A review of the results and conclusions from the OB Grounds LTM program will be provided in the 5-year LUC Review. As presented and summarized above, available monitoring data shows no evidence of total lead or total copper in the groundwater above the cleanup goals subsequent to the completion of the remedial action for the Site. These findings are consistent with the groundwater analytical results obtained during the remedial investigation stage (1990s) of work at the Site, indicating that there is no evidence of groundwater quality deterioration over approximately 20 years. Further, the annual inspections of the soil cover have shown minimal evidence of erosion or animal breaching of the protective soil cover.

The examination of spillways connecting the OB Grounds to Reeder Creek indicate that measures performed to eliminate overland soil transport from the OB Grounds to Reeder Creek continue to exist and have been effective, as there is no indication that soil or debris from the OB Grounds is located in the spillways downgradient of the control measures. Finally, the inspection of Reeder Creek indicates that the bedrock that underlies the watercourse adjacent to the OB Grounds continues to be scoured by the

perennial flow within the creek. Currently, there is minimal indication that sediment is being redeposited at locations from which it was previously excavated. Therefore, due to the absence of any evidence that suggests contaminants of concern have been mobilized from the OB Grounds either via the groundwater or overland flow of storm-event waters, and due to the continued scouring of the creek bed by the perennial flow of water, there is no reason to develop or implement a sediment monitoring plan for Reeder Creek at this time.

With mutual agreement of all parties upon acceptance of the OB Grounds LTM conclusions presented in the Five Year Review Report, no further LTM monitoring of the groundwater will occur at the OB Grounds. Soil cover inspections will continue and be performed as part of annual LUC inspections.

7.0 REFERENCES

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TABLES

Table 1
Site-Specific Cleanup Goals for Groundwater
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

ANALYTES	Action Level Water (µg/L)
Copper	200
Lead	15

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998 through addendum June 2004)
2. Lead action level is from USEPA Maximum Contaminant Limit (MCL):
www.epa.gov/safewater/mcl.html#inorganic.html

Table 2
Groundwater Elevation Data
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Monitoring Well	MW23-1		MW23-2		MW23-3		MW23-4		MW23-5		MW23-6	
Top of Riser Elevation (ft)	622.64		622.28		619.18		637.11		639.47		632.59	
Sampling Round - Date	Depth to Groundwater (ft)	Water Level Elevation (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)
Round 1 - November 20, 2007	12.00	610.64	9.60	612.68	10.80	608.38	8.60	628.51	7.00	632.47	8.35	624.24
Round 2 - February 25, 2008	11.46	611.18	8.78	613.50	9.24	609.94	3.20	633.91	2.85	636.62	3.78	628.81
Round 3 - May 20, 2008	11.63	611.01	9.17	613.11	9.68	609.50	4.14	632.97	5.19	634.28	5.54	627.05
Round 4 - August 25, 2008	12.10	610.54	9.84	612.44	10.59	608.59	7.82	629.29	8.33	631.14	10.08	622.51
Round 5 - August 02, 2010	12.06	610.58	9.40	612.88	9.97	609.21	5.81	631.30	7.51	631.96	8.79	623.80
Round 6 - October 03, 2011	11.57	611.07	6.84	615.44	9.31	609.87	4.47	632.64	5.22	634.25	9.48	623.11
Round 7 - October 08, 2012	11.94	610.70	9.34	612.94	10.65	608.53	9.41	627.70	9.09	630.38	10.73	621.86
Round 8 - December 09, 2013	11.36	611.28	7.72	614.56	7.93	611.25	3.04	634.07	2.84	636.63	3.79	628.80
Round 9 - October 14, 2014	12.15	610.49	9.96	612.32	9.95	609.23	6.62	630.49	7.42	632.05	8.80	623.79
Round 10 - October 19, 2015	12.02	610.62	8.65	613.63	9.32	609.86	4.54	632.57	5.64	633.83	6.67	625.92

Notes:

Historical Max: MW23-1: Round 8; MW23-2: Round 6; MW23-3: Round 8; MW23-4: Round 8; MW23-5: Round 8; MW23-6: Round 2

Historical Min: MW23-1: Round 9; MW23-2: Round 9; MW23-3: Round 1; MW23-4: Round 7; MW23-5: Round 7; MW23-6: Round 7

Table 3
Groundwater COC Results - Round 10
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Area								OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds	
Loc ID								MW23-1		MW23-2		MW23-2		MW23-3		MW23-4		MW23-5		MW23-6	
Matrix								GW		GW		GW		GW		GW		GW		GW	
Sample ID								OBLM20064		OBLM20065		OBLM20066		OBLM20067		OBLM20068		OBLM20069		OBLM20070	
Sample Date								10/21/2015		10/21/2015		10/21/2015		10/20/2015		10/20/2015		10/20/2015		10/20/2015	
QC Type								SA		SA		DU		SA		SA		SA		SA	
Study ID								LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round								10		10		10		10		10		10		10	
Parameter	**Unit**	**Maximum Value**	**Frequency of Detection**	**Action Level**	**Number of Exceedances**	**Number of Times Detected**	**Number of Samples Analyzed**	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Copper	UG/L	0	0%	200	0	0	7	1.8	U	1.8	U	1.8	U	1.8	U	1.8	U	1.8	U	1.8	U
Lead	UG/L	0	0%	15	0	0	7	3.9	U	3.9	U	3.9	U	3.9	U	3.9	U	3.9	U	3.9	U

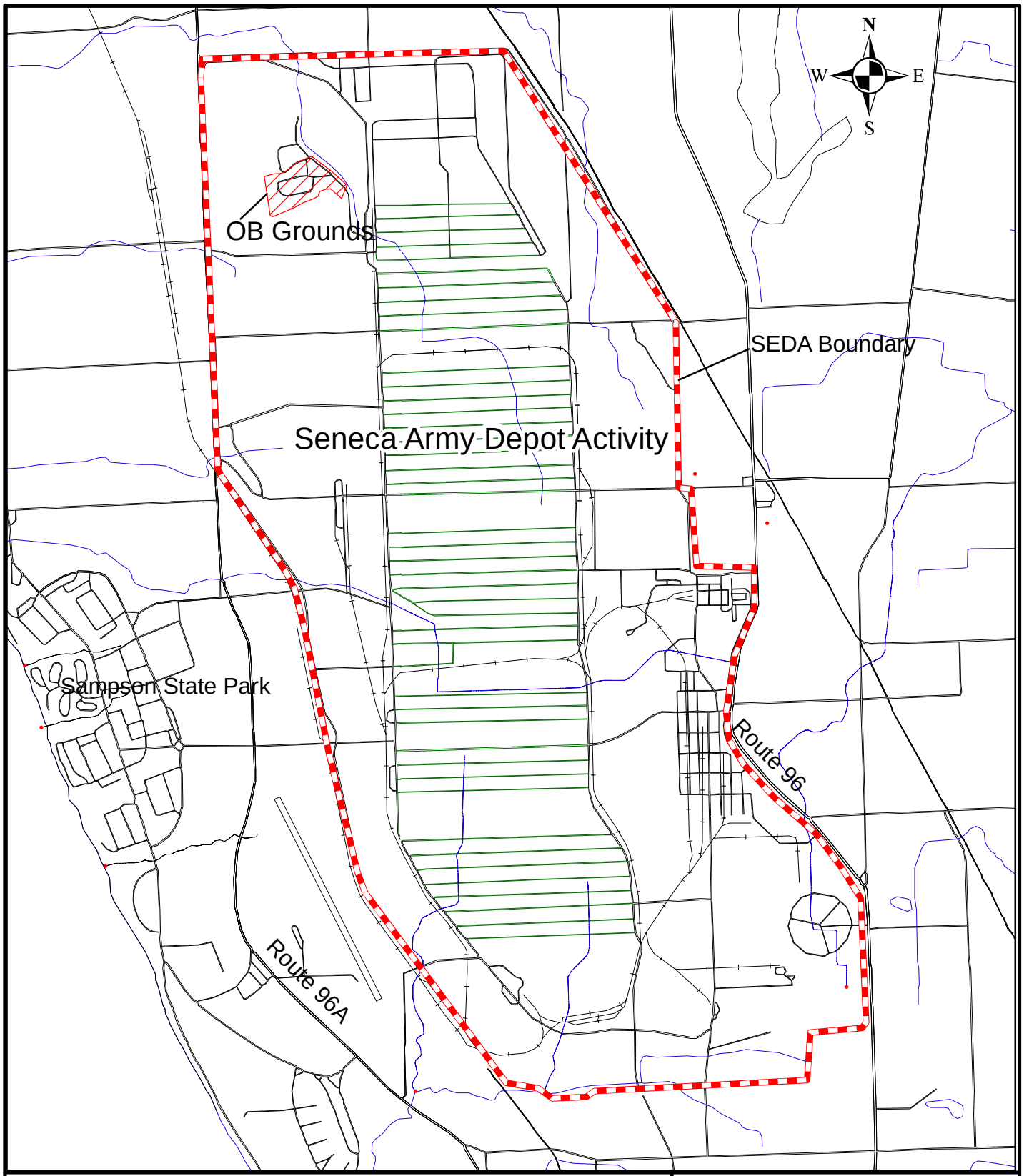
Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 10 samples were analyzed by SW846-6010C.
Qual = Qualifier
U = compound was not detected
J = the reported value is an estimated concentration
SA = Field Sample
DU = Field Sample Duplicate

Table 4
Soil Cover Inspection Log
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Grid #	Round 9 - October 2014	Round 10 - October 2015
A5	No animal holes were observed. Areas in the northeast corner of the grid are not as vegetated as the surrounding area. Same vegetation density as observed in previous years.	No animal holes were observed. Northeast corner of the grid has areas of sporadic vegetation. Same vegetation density as observed in previous years.
B3	No animal holes were observed. Standing water in multiple locations. Thick vegetation throughout.	No animal holes or areas with standing water were observed. Area was very well vegetated.
C7	No animal holes observed. Shallow tire ruts (2-3" deep) which did not impact the underlying soil cap. Area was regraded to smooth out ruts and covered with shale.	No animal holes observed. Shale used to fill in tire ruts observed in 2014 still in place. Area is very well vegetated.
D7	No animal holes were observed. Standing water in area of grid. Not as well vegetated as surrounding grids.	Sporadic vegetation, not as densely vegetated as the surrounding area. Same vegetation density as observed in previous years.
I6	No animal holes were observed. Minor surface water erosion along the northern edge of the cap. Well vegetated.	No animal holes were observed. Similar to past inspections, minor surface water erosion along the northern edge of the cap was observed. Well vegetated.
I7	No animal holes were observed. Minor surface water erosion along the western edge of the cap. Thin vegetation cover along edge of grid.	No animal holes were observed. The area was well vegetated and similar to past inspections, minor surface water erosion was observed along the western edge of the cap.
I8	No animal holes were observed. Previously observed sporadic vegetation along the western edge of the grid extending 3-4 feet into the grid. No change in previously observed run off conditions.	No animal holes were observed. Previously observed sporadic vegetation along the southern and western edge of the grid extending 3-4 feet into the grid. No change in previously observed run off conditions.
J6	No animal holes were observed. Thin vegetation.	No animal holes were observed. Well vegetated surrounding the road.
J8	No animal holes were observed. Standing water surrounding the grid. Well vegetated.	No animal holes were observed. The area was well vegetated and no standing water was observed.
L8	No animal holes were observed. Standing water on each side of road. Little vegetation on sides of road.	No animal holes were observed. Little vegetation on sides of road.

Notes:
1. All grids capped areas were inspected. Grids with no signs of erosion or other disturbances to the cover are not included in this log.
2. Standing water in capped areas during the 2014 inspection was the result of steady rainfall that occurred on the day of the inspections.

FIGURES



Approximate Boundary of SEDA Site

Approximate Boundary and extent of OB Grounds

0 2,500 5,000 10,000 15,000 Feet

PARSONS

CLIENT / PROJECT TITLE

SENECA ARMY DEPOT

OPEN BURNING (OB) GROUNDS

LTM 2015 ANNUAL REPORT

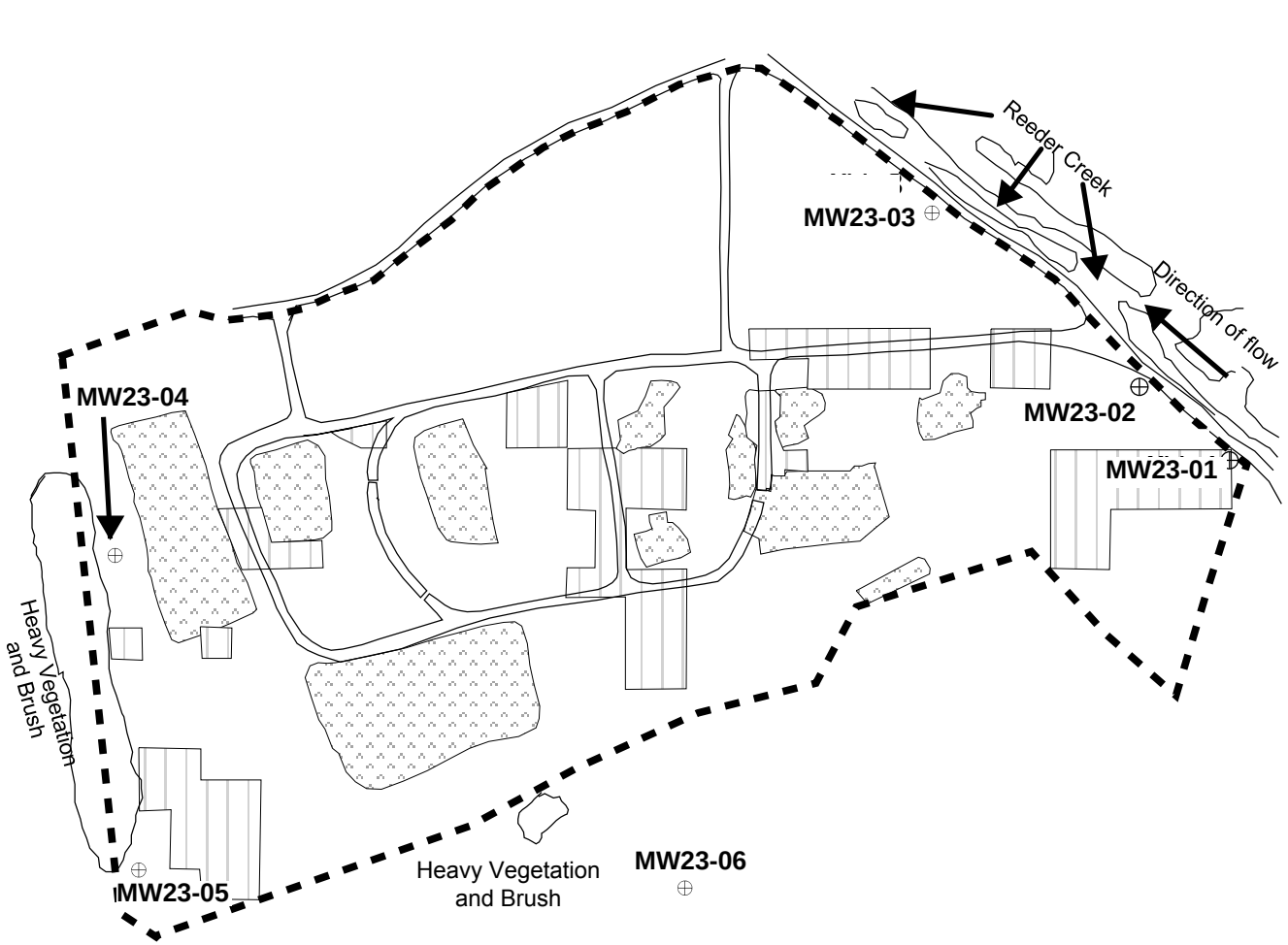
DEPT: ENVIRONMENTAL REMEDIATION

Figure 1

SEDA Site Map and AOC Location

EDITED BY TDV | DATE DECEMBER 2015

Path: O:\Seneca\Depot Survey\SEDA_Site_Map-OB_Grounds.mxd



Interred Soils

Former Burning Pads

OB Grounds Boundary

Existing Monitoring Wells

Notes:
(1) Map is not to scale. Location of features shown are approximate.
(2) Map is based on information presented on Figure 4.13 of *Soil and Sediment Remediation, Open Burning Grounds, Completion Report* (Weston Solutions Inc., June 2005).

PARSONS

SENECA ARMY DEPOT
OPEN BURNING (OB) GROUNDS
LTM 2015 ANNUAL REPORT

Figure 2
Open Burning Grounds Site Map

Date: December 2015

"P:\PIT\Projects\Huntsville Cont W912DY-08-D-0003\TO#15 - LTM and LUC\OB Grounds LTM FY 13\LTM 2013 Annual Report\Draft\Figures\PDF\Figure 2 OB Grounds Site Map.pdf"

APPROXIMATE LOCATION OF GROUNDWATER DIVIDE (BASED ON APRIL 1993 GW ELEVATION DATA)

REEDER CREEK

N

MW23-3 609.86

MW23-2 613.63

MW23-1 610.62

MW23-4 632.57

MW23-6 625.92

MW23-5 633.83

W-1 W-2 W-3 W-4 W-5 W-6 W-7 W-8 W-9 W-10 W-11 W-12 W-13 W-14 W-15 W-16 W-17 W-18 W-20 W-21 W-22 W-23 W-24 W-25 W-26 W-27 W-28 W-29 W-30 W-31 W-32 W-33 W-34 W-35 W-36 W-37 W-38

MW-1 627.92
MW-2 619.42
MW-3 (619.55)
MW-4 616.75
MW-5 635.05
MW-6 626.91
MW-7 619.90
MW-8 636.01
MW-9 630.55
MW-10 636.40
MW-11 631.19
MW-12 623.12
MW-13 625.21
MW-14 622.15
MW-15
MW-16 620.61
MW-17 623.08
MW-18 621.97
MW-19 634.15
MW-20 617.10
MW-21 634.83
MW-22 620.81
MW-23
MW-24 622.74
MW-25 620.53
MW-26 620.81
MW-27
MW-28 616.29
MW-29 628.42
MW-30 624.37
MW-31 634.13
MW-32 632.39
MW-33 628.19
MW-34 634.86
MW-35 628.34
MW-36
MW-37
MW-38
MW-39
MW-40
MW-41 625.45
GB-21
SEAD-4

LEGEND:

- G — BURNING PAD DESIGNATION
- SEAD-4 — SURVEY MONUMENT
- 650 — TOPOGRAPHICAL CONTOURS
- W-1 — WETLAND & DESIGNATION
- 625.92 — CURRENT MONITORING WELL LOCATION WITH OCTOBER 2015 LTM GAUGING DATA
- 611.01 — HISTORICAL MONITORING WELLS WITH APRIL 1993 DATA
- 618 — HISTORIC GROUNDWATER ELEVATION CONTOUR (APRIL 1993) MSL DATUM
- GENERAL GROUNDWATER FLOW DIRECTION
- APPROXIMATE BOUNDARY AND EXTENT OF OB GROUNDS

100 0 100 200
1" = 200'

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT ACTIVITY
OPEN BURNING (OB GROUNDS)
LTM 2015 ANNUAL REPORT

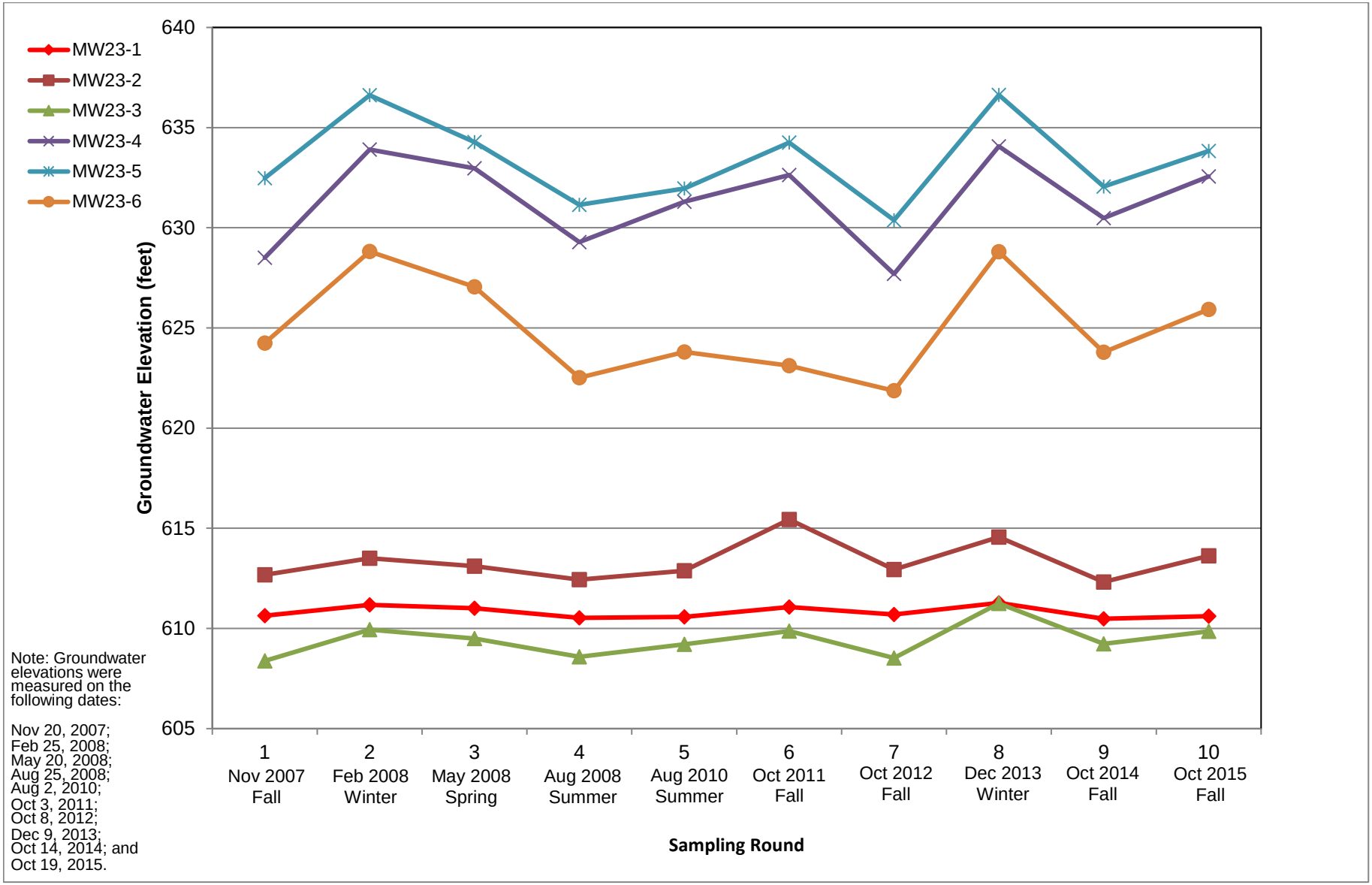
DEPT.	PROJECT. No.
ENVIRONMENTAL ENGINEERING	748662-01600

Figure 3
Historic Groundwater Contours and October 2015 Groundwater Elevations

SCALE	DATE	REV
1" = 200'	DECEMBER 2015	-

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\OB GROUNDS LTM FY 15\LTM 2015 ANNUAL REPORT\DRAFT\FIGURES\FIGU, DATE: 12/14/2015 01:47:37PM, p0018397

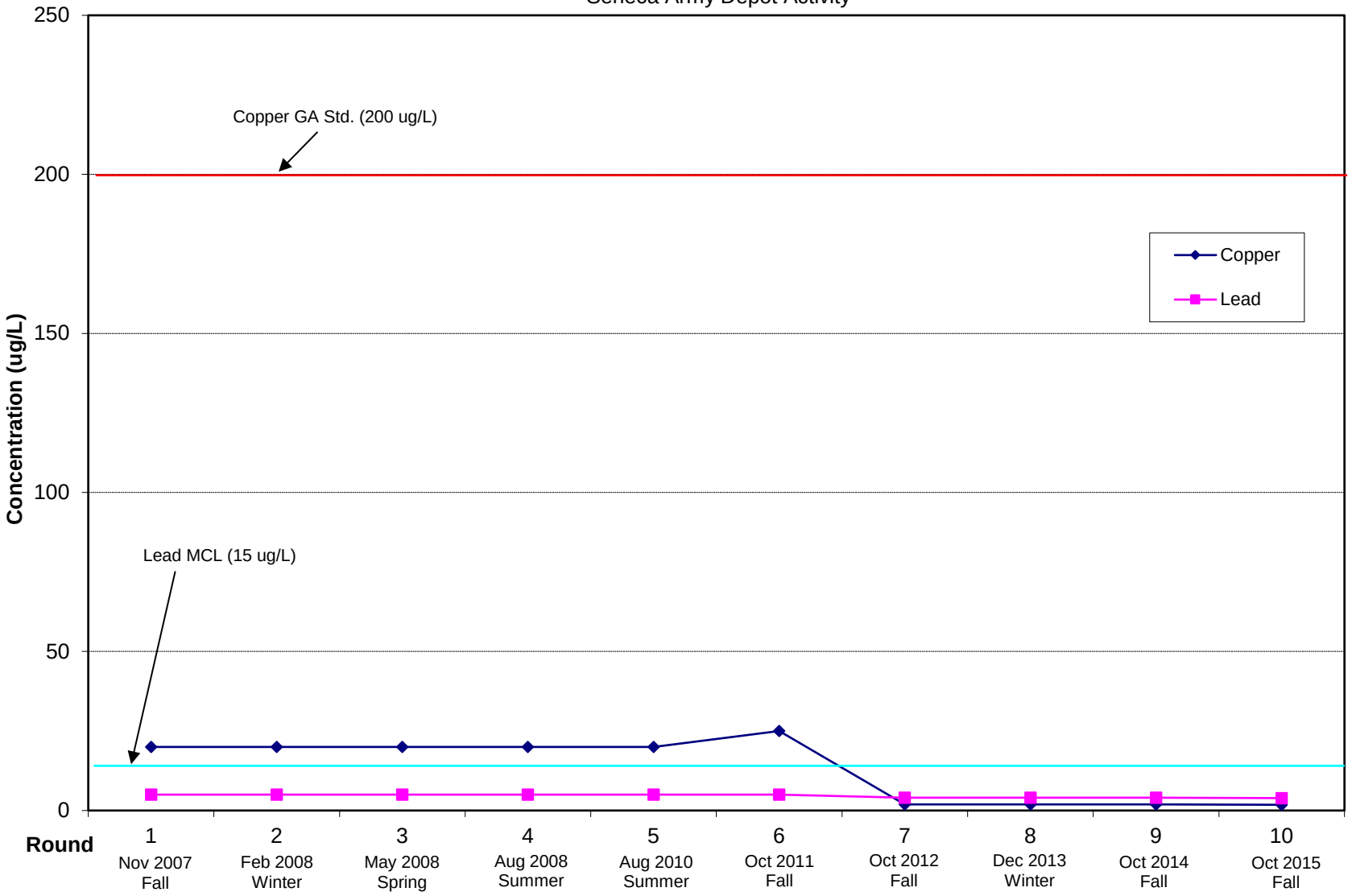
Figure 4
Groundwater Elevation Profiles
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity



Note: Groundwater elevations were measured on the following dates:

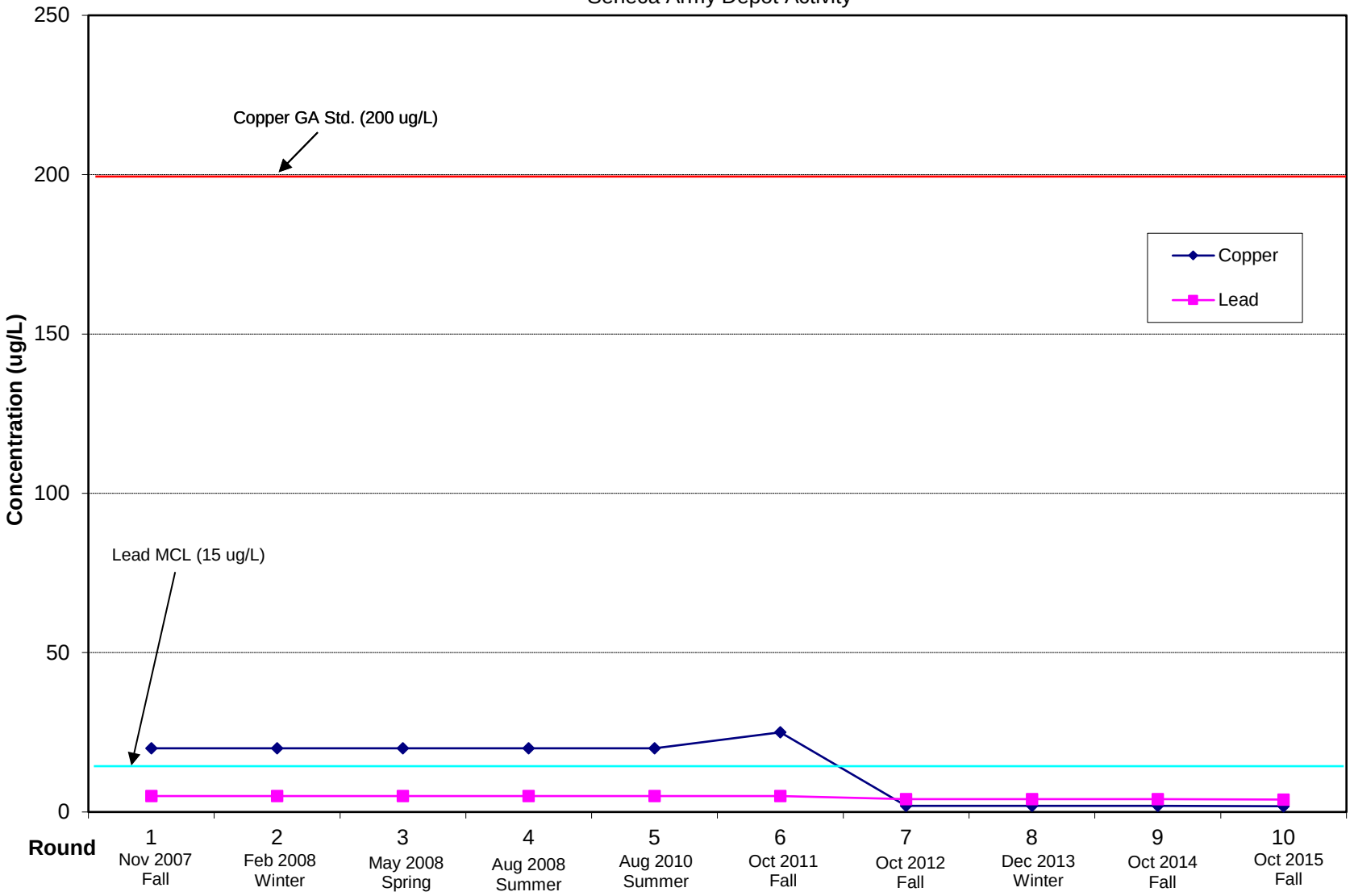
Nov 20, 2007;
Feb 25, 2008;
May 20, 2008;
Aug 25, 2008;
Aug 2, 2010;
Oct 3, 2011;
Oct 8, 2012;
Dec 9, 2013;
Oct 14, 2014; and
Oct 19, 2015.

Figure 5
Concentrations of Total Lead and Total Copper at MW23-1
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity



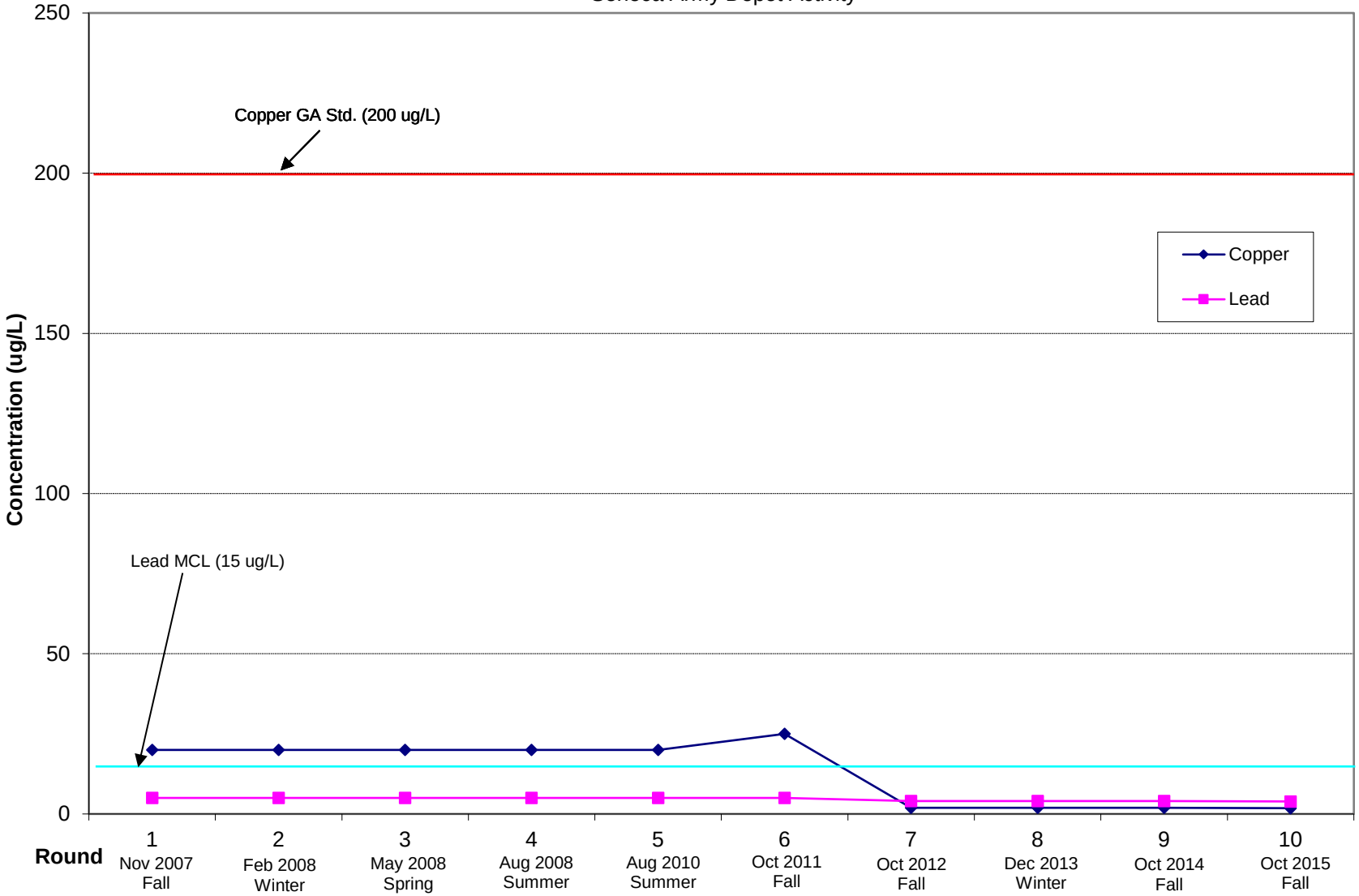
Notes: Groundwater samples were collected on the following dates: Nov 21, 2007; Feb 25, 2008; May 21, 2008; Aug 26, 2008; Aug 2, 2010; Oct 3, 2011; Oct 9, 2012; Dec 10, 2013; Oct 16, 2014; and Oct 21, 2015.
Groundwater sampling was performed quarterly through August 2, 2010, and annually thereafter. Total copper and total lead concentrations in groundwater were below detection limits.

Figure 6
Concentrations of Total Lead and Total Copper at MW23-2
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity



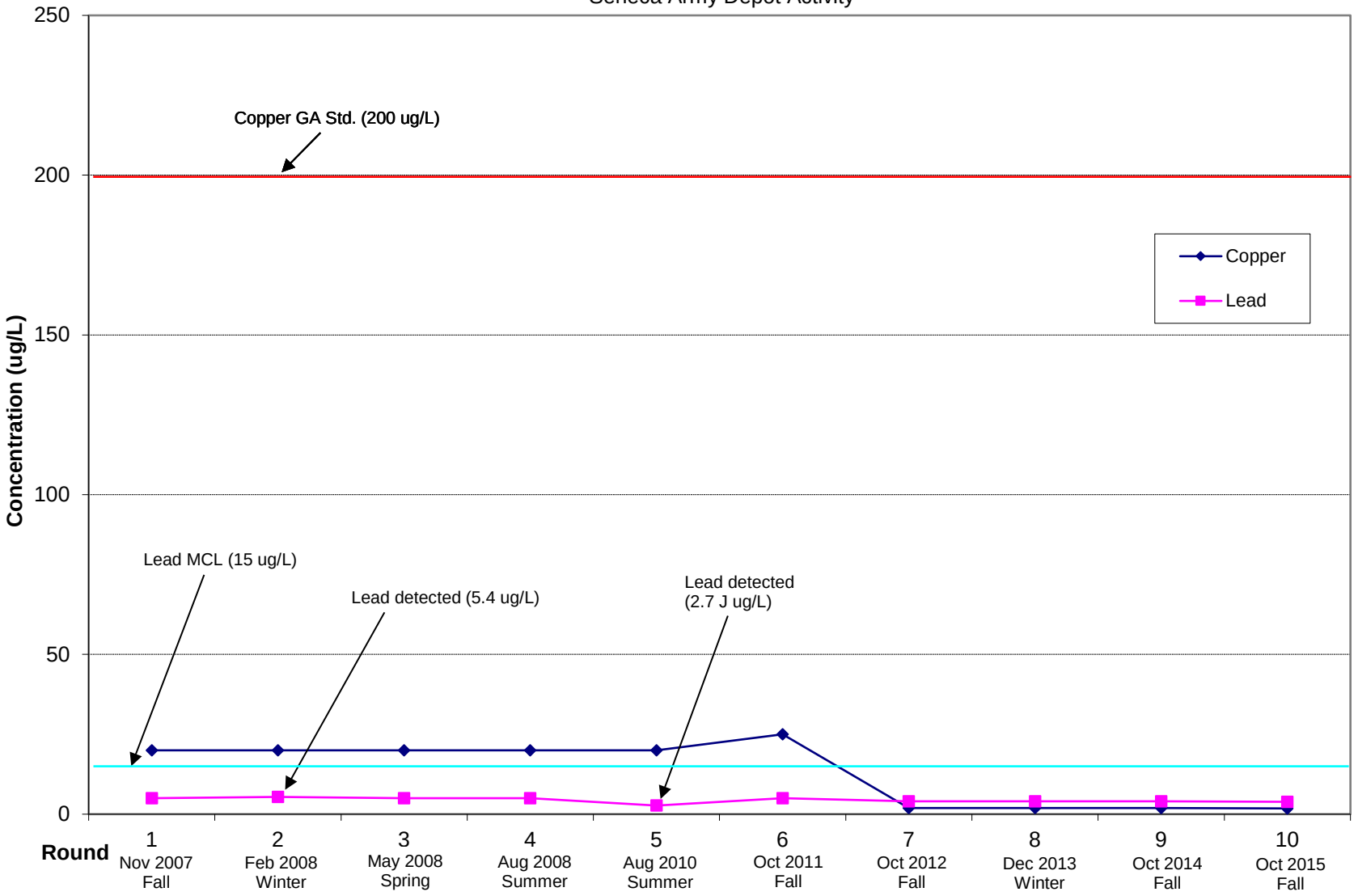
Notes: Groundwater samples were collected on the following dates: Nov 21, 2007; Feb 25, 2008; May 21, 2008, Aug 26, 2008; Aug 2, 2010; Oct 3, 2011; Oct 9, 2012; Dec 11, 2013; Oct 16, 2014; and Oct 21, 2015.
Groundwater sampling was performed quarterly through August 2, 2010, and annually thereafter. Total copper and total lead concentrations in groundwater were below detection limits.

Figure 7
Concentrations of Total Lead and Total Copper at MW23-3
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity



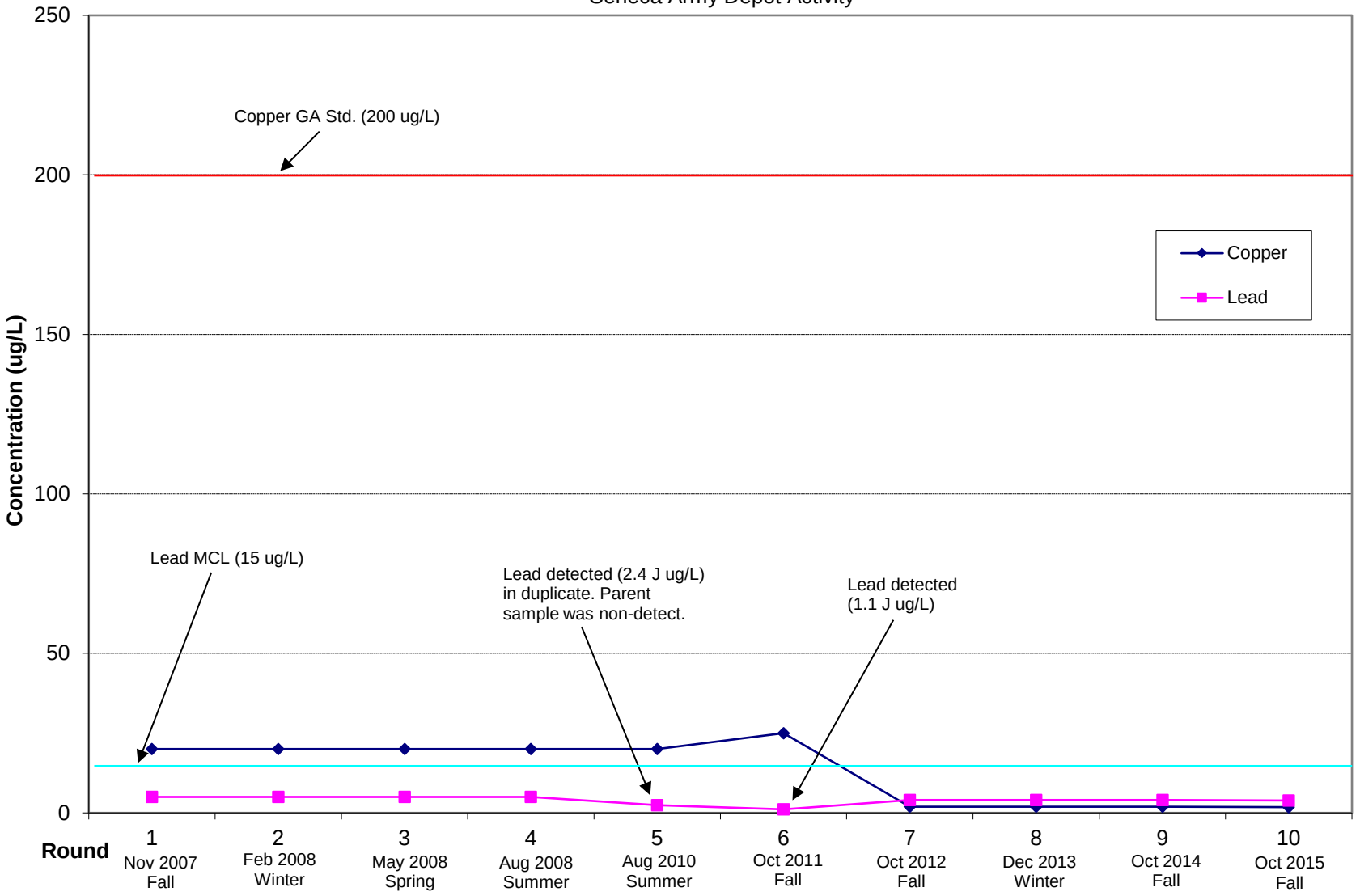
Notes: Groundwater samples were collected on the following dates: Nov 21, 2007; Feb 25, 2008; May 21, 2008; Aug 26, 2008; Aug 2, 2010; Oct 3, 2011; Oct 8, 2012; Dec 10, 2013; Oct 16, 2014; and Oct 20, 2015.
Groundwater sampling was performed quarterly through August 2, 2010, and annually thereafter. Total copper and total lead concentrations in groundwater were below detection limits.

Figure 8
Concentrations of Total Lead and Total Copper at MW23-4
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity



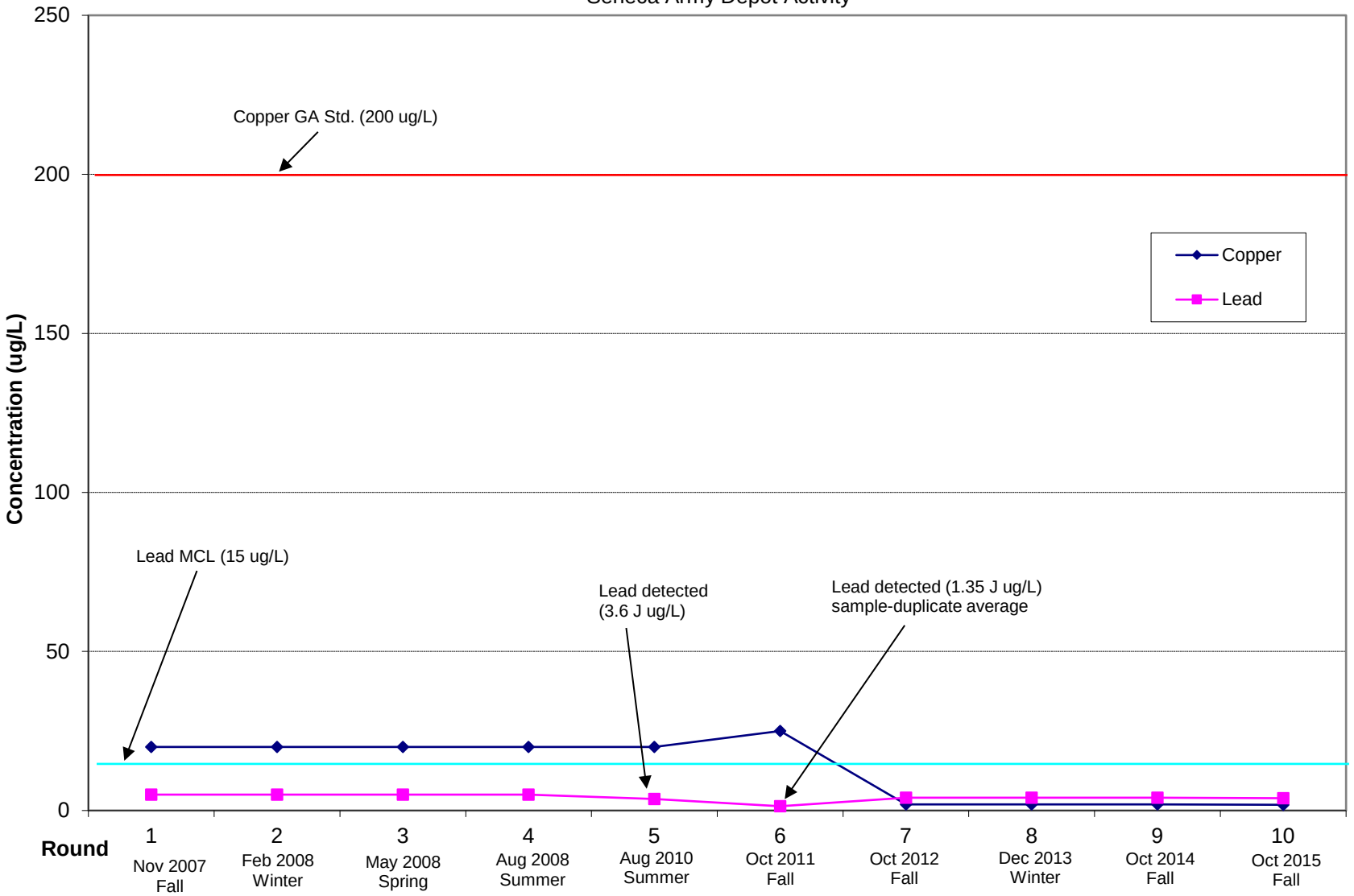
Notes: Groundwater samples were collected on the following dates: Nov 21, 2007; Feb 25, 2008; May 21, 2008; Aug 26, 2008; Aug 2, 2010; Oct 3, 2011; Oct 8, 2012; Dec 10, 2013; Oct 15, 2014; Oct 20, 2015; and Oct 20, 2015.
Groundwater sampling was performed quarterly through August 2, 2010, and annually thereafter. Total copper and total lead concentrations in groundwater were below detection limits except where otherwise noted.

Figure 9
Concentrations of Total Lead and Total Copper at MW23-5
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

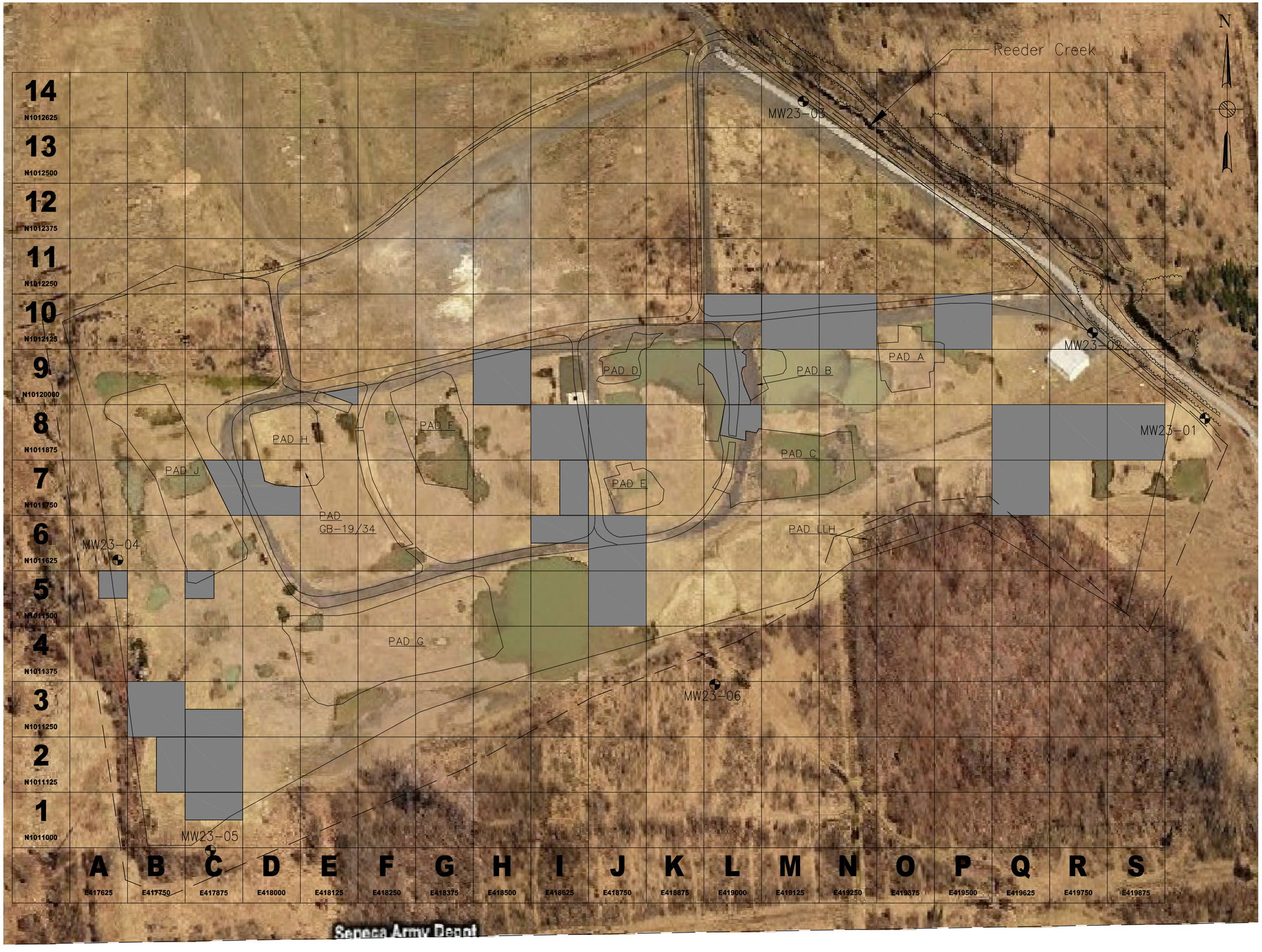


Notes: Groundwater samples were collected on the following dates: Nov 21, 2007; Feb 25, 2008, May 21, 2008; Aug 26, 2008; Aug 2, 2010; Oct 3, 2011; Oct 8, 2012; Dec 10, 2013; Oct 15, 2014; and Oct 20, 2015. Groundwater sampling was performed quarterly through August 2, 2010, and annually thereafter. Total copper and total lead concentrations in groundwater were below detection limits except where otherwise noted.

Figure 10
Concentrations of Total Lead and Total Copper at MW23-6
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity



Notes: Groundwater samples were collected on the following dates: Nov 21, 2007; Feb 25, 2008; May 21, 2008; Aug 26, 2008; Aug 2, 2010; Oct 3, 2011; Oct 8, 2012; Dec 10, 2013; Oct 16, 2014; and Oct 20, 2015. Groundwater sampling was performed quarterly through August 2, 2010, and annually thereafter. Total copper and total lead concentrations in groundwater were below detection limits except where otherwise noted.



LEGEND

- WELLS INSTALLED AUGUST 2007
- AREA OF 9-INCH SOIL COVER
- — — APPROXIMATE BOUNDARY AND EXTENT OF OB GROUNDS

NOTES:

1. THE SOIL COVER AND GRID LOCATIONS WERE PROVIDED BY WESTON SOLUTIONS, INC. (JUNE 2005)
2. THE GRID SYSTEM IS OVERLAYED OVER AN AERIAL IMAGE OF THE SITE. FIGURE NOT TO SCALE.

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT
OPEN BURNING (OB) GROUNDS
LTM 2015 ANNUAL REPORT

DEPT.	Dwg. No.
ENVIRONMENTAL ENGINEERING	748662-01600

Figure 11
OB Grounds
Soil Cover Areas and Well Locations

SCALE	DATE	REV
N.T.S.	December 2015	–

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\OB GROUNDS LTM FY 15\LTM 2014 ANNUAL REPORT\DRAFT\FIGURES\FIGU, DATE: 11/10/2014 12:44:48PM, p0018397

0 50 100 200 300 400 Feet

N

Beaver Dam Location
(observed 2013 & 2014)

Reeder Creek

MW23-3

MW23-2

MW23-1

0 37.5 75 150 225 300 Feet

N

Approx. downstream limits of excavated creek bottom and beginning of creek inspection

0 125 250 500 750 1,000 Feet

Direction of stream flow

OD Grounds (SEAD-45)

OB Grounds (SEAD-23)

Approx. upstream limits of excavated creek bottom and end of creek inspection

Legend

1 Photo ID with directional arrow. Numbers without arrow, view is down

MW23-1 Monitoring Well

OB Grounds Boundary

Stagnant Pooling Area

Vegetation Embankment

OD Grounds 2012 Creek Brush Cutting Area

PARSONS

SENECA ARMY DEPOT ACTIVITY
Open Burning Grounds
LTM 2015 Annual Report

FIGURE 12
Reeder Creek Inspection
Photo Locations (October 19, 2015)

December 2015	Ver 1.0	TIB

Path: P:\PIT\Projects\Huntsville Cont W912DY-08-D-0003\TO#15 - LTM and LUC\OB Grounds LTM FY 15\LTM 2015 Annual Report\Draft\Figures\Figure 12 Creek Photo Loc_2015.mxd

APPENDICES

A	Open Burning Grounds Long-Term Monitoring Round 10 Field Forms
B	Complete Groundwater Monitoring Results for OB Grounds LTM
C	Laboratory Reports (provided on the electronic (CD) version of this report)
D	Data Validation Report
E	Reeder Creek Inspection Photos (October 2015)
F	Statistical Analysis of LTM Results
G	Soil Cap Inspection Photo Log (October 2015)

APPENDIX A

OPEN BURNING GROUNDS LONG-TERM MONITORING ROUND 10 FIELD FORMS

GROUNDWATER ELEVATION REPORT

PARSONS	CLIENT:	DATE: 10/19/2015
PROJECT: OB GROUNDS LTM - ROUND 10		PROJECT NO:
LOCATION: SEDA, Romulus, NY		INSPECTOR: BBO + TDV

MONITORING EQUIPMENT:

INSTRUMENT	DECTECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR:

INSTRUMENT	CORRECTION FACTOR

COMMENTS: 41°F and Sunny

WELL	TIME	DEPTH TO WATER	DEPTH TO ~~PRODUCT~~ Well Bottom	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC. GRAV.	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of: riser, concrete, protective casing, etc.)
MW23-1	1145	12.02	15.11					locked
MW23-2	1140	8.65	15.10					locked
MW23-3	1135	9.32	14.77					locked
MW23-4	1105	4.54	17.75					locked, concrete casing lifted out of box
MW23-5	1115	5.64	17.52					locked, concrete casing lifted out of box
MW23-6	1126	6.67	17.51					locked, concrete casing lifted out of box
								*Due to rust concerns, all locks were replaced

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW23-1

PROJECT: OB Grounds LTM Groundwater Sampling - Round 10
LOCATION: ROMULUS, NY

DATE: 10/21/15
INSPECTORS: TV
PUMP #: 7959 - peristaltic
SAMPLE ID #: OBLM20064

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
0903	58	Cloudy		5	SE	Dry	OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
HISTORIC DATA	15.11					

	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
DATA COLLECTED AT WELL SITE		12.0			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
0909	12.11	124		YSI 6122		Horiba 1973			Hach 4785
0911	12.19	118		1.43	15.5	0.696	6.99	238	4.87
0916	12.17	86		0.40	15.4	0.691	6.79	244	3.42
0921	12.17	104		0.25	15.4	0.670	6.77	241	3.40
0926	12.19			0.35	15.4	0.688	6.73	242	3.14
0931	12.20			0.51	15.4	0.688	6.74	236	2.81
0936	12.20			0.66	15.3	0.687	6.76	229	2.36
0941	12.22			0.45	15.3	0.687	6.80	202	1.42
0946	12.22		~1 gal	0.54	15.3	0.688	6.95	168	1.26
0951	12.22			0.50	15.3	0.689	6.93	128	1.35
0956	12.20	106		0.28	15.3	0.691	6.92	99	0.92
1001	12.19			0.52	15.3	0.689	6.88	73	1.02
1006	12.18		~2 gal	0.43	15.3	0.687	6.88	66	0.98
1011	12.19			0.39	15.3	0.689	6.88	60	0.90
1016	12.19		~2.5 gal	0.39	15.3	0.690	6.94	54	0.77
1022			collect sample	1 250mL bottle					
1033	12.20		post-sample ~3 gal	0.43	15.3	0.694	6.94	62	1.09

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW23-2
PROJECT: OB Grounds LTM Groundwater Sampling - Round 10		DATE: 10/21/15
LOCATION: ROMULUS, NY		INSPECTORS: BDO
		PUMP #: Parsen Peristaltic
		SAMPLE ID #: OBLM20065/66

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
905	57	Partly sunny		S-7N	0-5	dry	OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

3 well Vol = 3.06 gal

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	15.10					
DATA COLLECTED AT WELL SITE	**PID READING (OPENING WELL)**	**DEPTH TO STATIC WATER LEVEL (TOC)**		**DEPTH TO STABILIZED WATER LEVEL (TOC)**	**DEPTH TO PUMP INTAKE (TOC)**	**PUMPING START TIME**
		8.83'				
RADIATION SCREENING DATA	**PUMP PRIOR TO SAMPLING (cps)**			**PUMP AFTER SAMPLING (cps)**		

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
917	8.70	YSI & tubing in	well YSI	YSI	Hach	Hach	Hach	Hach	
917		Pump Started							
924	9.52	142		0.24	15.5	0.688	6.94	219	7.97
929	9.61	120		0.22	15.6	0.683	6.97	216	3.27
934	9.69			0.17	15.7	0.676	6.97	211	2.24
945 939	9.80			0.32	15.7	0.670	6.97	201	1.73
950	9.86	~140	0.75 gal	0.43	15.8	0.673	6.97	197	2.22
955	9.89			0.40	15.7	0.675	6.95	191	2.01
1000	9.93			0.29	15.7	0.674	6.97	189	1.09
1005	9.97	144	~1.25	0.28	15.7	0.672	6.96	186	1.29
1010	10.04			0.31	15.7	0.671	6.96	185	0.97
1015	10.13		~1.75	0.26	15.7	0.669	6.97	182	0.99
1020	10.10	155		0.26	15.7	0.668	6.97	180	0.78
1025	10.14		~2.0	0.18	15.7	0.667	6.98	178	0.92
1030	10.15			0.17	15.7	0.668	6.98	177	0.98
1035	10.15		~2.5	0.14	15.7	0.667	6.98	176	0.92
1040	10.14	158	~2.75	0.12	15.6	0.665	6.98	176	1.02
1045	10.18		~3.0	0.13	15.6	0.662	6.98	175	0.99
									Time
1052		Sample Collected		4x Plastic 250ml bottles				Dup	1056

1102 Restarted Pump to Collect Post-Sample Collection Geo Parameters

1107 10.05 ~3.25 gals 0.11 15.6 0.658 7.60 179

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW23-3

PROJECT: OB Grounds LTM Groundwater Sampling - Round 10
LOCATION: ROMULUS, NY

DATE: ~~MW23-3~~ TV 10/20/2015
INSPECTORS: BBO + TV
PUMP #: Parsons Peristaltic
SAMPLE ID #: OBLM20067

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	(FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
1408	63	Cloudy		5	SW	Dry	OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	14.77					
DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)		DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		9.36				
RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)			PUMP AFTER SAMPLING (cps)		

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
1424	9.35			YSI 2854		Horiba 9761			Hach 4785
~~1429~~									
1431	9.44	140		0.04	15.0	0.747	7.17	-28	4.58
1435	9.45	122		0.06	15.0	0.744	7.15	-18	3.33
1440	9.45	98		0.09	15.0	0.742	7.15	-17	1.78
1445	9.44	102		0.10	15.0	0.741	7.15	-15	1.24
1450	9.44			0.11	14.9	0.741	7.15	-15	1.46
1455	9.44		~1 gal	0.13	14.9	0.742	7.14	-14	0.80
1500	9.44			0.14	14.9	0.741	7.15	-15	0.80
1505	9.44			0.16	14.9	0.737	7.15	-15	0.57
1510	9.45	108		0.17	14.9	0.738	7.14	-15	0.51
1515	9.45			0.18	14.9	0.737	7.13	-14	0.76
1520	9.45			0.19	14.9	0.737	7.14	-15	0.35
1525	9.45			0.20	14.9	0.735	7.12	-14	0.62
1530	9.45		~1.75 gal	0.20	14.9	0.736	7.14	-14	0.57
1535			sample collected	1-250mL	bottle				
1545	9.43		post-sample	0.22	14.9	0.735	7.15	-12	0.95
			~2.0 gal						

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW23-4

PROJECT: OB Grounds LTM Groundwater Sampling - Round 10
LOCATION: ROMULUS, NY

DATE: 10/20/2015
INSPECTORS: ~~Parsons Per~~ BBO + TV
PUMP #: Parsons Peristaltic pump
SAMPLE ID #: OBLM20068

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
1225	59	~~59~~ overcast		5	SW		OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	~~4.50~~ TV 17.75'					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		4.50'			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
1229	4.39			YSI 2854		Horiba 9761			Hach 4785
1240	5.71	100		0.05	14.9	0.798	7.93	166	22.6
1245	6.39			0.21	14.9	0.779	7.89	161	9.86
1250	6.86			0.19	15.0	0.794	7.84	154	6.12
1255	7.41			1.57	15.1	0.779	7.82	146	3.80
1300	7.85			1.53	15.1	0.761	7.85	142	2.48
1305	8.39	120	~1 gal	1.63	15.1	0.749	7.85	141	2.97
1310	8.77			1.63	15.1	0.746	7.88	140	2.88
1315	9.03	105		1.63	15.1	0.744	7.89	141	1.76
1320	9.33			1.67	15.1	0.743	7.87	144	1.31
1325	9.19			1.62	15.1	0.741	7.86	147	1.30
1330	9.32			1.51	15.0	0.739	7.86	152	1.52
1335	9.52			1.49	15.0	0.741	7.80	153	1.50
1340	9.75		~2 gal	1.50	15.0	0.741	7.80	155	1.11
1346			sample collected	1-250mL	bottle				
1348		Restarted Pump to collect Post-Sample Geo Parsons							
1351	10.05		~2.25 gal	1.29	15.1	0.744	7.74	156	1.73

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW23 - 5

PROJECT: OB Grounds LTM Groundwater Sampling - Round 10
LOCATION: ROMULUS, NY

DATE: 10/20/2015
INSPECTORS: BBO + TV
PUMP #: Parsons Peristaltic pump
SAMPLE ID #: OBLM20069

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
0915	56	overcast		5	SW	Dry	OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	17.52					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		5.77			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
0928	5.65	116		YSI 2854		Horiba 29761			Hach 4785
0934	6.71			0.29	13.6	0.769	7.34	210	1.96
0939	6.90			0.22	13.7	0.755	7.32	200	2.24
0944	7.05			0.26	13.7	0.738	7.30	203	1.71
0950	7.21			0.38	13.7	0.730	7.29	206	1.75
0955	7.25			0.40	13.7	0.719	7.30	208	1.52
1000	7.18			0.42	13.7	0.713	7.30	209	1.38
1005	7.11	90	~1.0	0.44	13.7	0.712	7.30	215	0.81
1010	7.10	98		0.43	13.7	0.710	7.31	211	0.56
1015	7.15	118		0.43	13.7	0.709	7.31	211	0.79
1020	7.20			0.38	13.7	0.706	7.27	211	1.60
1025	7.26			0.32	13.7	0.704	7.31	205	0.27
1031		Collected Sample							
		Filled 1x Plastic		bottle					
			250mL						

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW23-6

PROJECT: OB Grounds LTM Groundwater Sampling - Round 10
LOCATION: ROMULUS, NY

DATE: 10/20/2015
INSPECTORS: BBO + TV
PUMP #: Parsons Peristaltic Pump
SAMPLE ID #: OBLM20070

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
1055	58	overcast		5	SW	Dry	OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	17.51					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		6.48			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
1105	6.48			YSI 2854		Horiba 9761			Hach 4785
1110	7.60	135		0.34	13.8	0.880	7.28	200	6.08
1115	8.34	100		0.30	14.0	0.886	7.24	171	4.95
1120	8.92			0.23	14.1	0.886	7.26	161	3.21
1125	9.36			0.14	14.3	0.888	7.27	136	2.32
1130	10.23	114	~1/2 gal	0.13	14.3	0.885	7.27	119	2.38
1135	10.80	90		0.21	14.3	0.885	7.26	127	1.37
1140	11.29	110		0.39	14.4	0.879	7.27	125	2.01
1145	11.89		1.0 gal	0.39	14.3	~~0.878~~ 0.878	7.29	126	1.12
1150	12.44			0.53	14.4	0.872	7.30	131	1.63
1154			sample collected	1-250mL bottle					
1155			Restart pump						
1200	12.85		post-sample	0.87	14.4	0.856	7.32	144	1.30
			~1.25 gal	~~0.89~~	~~14.4~~				

OB Grounds
Task Order #15
Round 10 Inspection

Date of Inspection: 10/19/2015
Weather Conditions: 46°, clear + sunny

Observations should include assessment of integrity of 9-inch soil cap placed over residual lead contaminated soil in 25 125'x125' grids.

Assessment should be made with respect to caps ability to ensure that indegenous terrestrial wildlife are not exposed via direct dermal contact or incidental ingestion.

Note signs of erosion or animal burrowing to ensure underlying soils are not exposed to the environment.

	Grid No.	Observations/Location of Disturbed Soils
1	A5	NE corner has areas of sporadic vegetation. No different from previous year's observations
2	C5	N/A; vegetated
3	B3	N/A; very well vegetated
4	B2	N/A; " " "
5	C3	N/A; " " "
6	C2	N/A; " " "
7	C1	N/A; " " "
8	C7	N/A, shale still in place used to fill tire ruts in 2014, very well vegetated
9	D7	N/A, same as previous years, sporadic vegetation, not as densely vegetated as surrounding areas.
10	E9	N/A; very well vegetated.
11	H9	N/A; well vegetated.
12	I6	N/A; " "
13	I7	N/A; very well vegetated
14	I8	No change in previously observed sporadic vegetation (Sand W edges), No change in previously observed runoff ditch
15	J5	N/A; very well vegetated
16	J6	N/A; very well vegetated
17	J8	N/A; Well vegetated

OB Grounds
Task Order #15
Round 10 Inspection

	Grid No.	Observations/Location of Disturbed Soils
18	L8	N/A, Vegetation on each side of the road.
19	L9	N/A; " " " " " " "
20	L10	N/A; Well vegetated
21	M10	N/A; Well vegetated
22	N10	N/A; Well vegetated
23	P10	N/A; Very well vegetated
24	Q7	N/A; Very well vegetated
25	Q8	N/A; " " "
26	R8	N/A; " " "
27	S8	N/A; " " "

APPENDIX B

COMPLETE GROUNDWATER MONITORING RESULTS FOR OB GROUNDS LTM

Appendix B
Complete Groundwater Monitoring Results for OB Grounds LTM
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

									Area	OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds	
									Loc ID	MW23-1		MW23-1		MW23-1		MW23-1		MW23-1		MW23-1	
									Matrix	GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	
									Sample ID	OBLM20001		OBLM20008		OBLM20009		OBLM20015		OBLM20022		OBLM20029	
									Sample Date	11/21/2007		2/26/2008		2/26/2008		5/21/2008		8/26/2008		8/3/2010	
									QC Type	SA		SA		DU		SA		SA		SA	
									Study ID	LTM		LTM		LTM		LTM		LTM		LTM	
									Sample Round	1		2		2		3		4		5	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value	Q	Value	Q	Value	Q	Value	Q	Value	Q	Value	Q
Inorganics																					
Copper	UG/L	0	0%	GA	200	0	0	70		20	U	20	U	20	U	20	U	20	U	20	U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		5	U	5	U	5	U	5	U	5	U	1.87	U

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
Q = Qualifier
U = compound was not detected
J = the reported value is an estimated concentration
SA = Field Sample
DU = Field Sample Duplicate

Appendix B
Complete Groundwater Monitoring Results for OB Grounds LTM
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

									Area	OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds	
									Loc ID	MW23-1		MW23-1		MW23-1		MW23-1		MW23-1		MW23-2	
									Matrix	GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	
									Sample ID	OBLM20036		OBLM20043		OBLM20050		OBLM20057		OBLM20064		OBLM20002	
									Sample Date	10/5/2011		10/9/2012		12/10/2013		10/16/2014		10/21/2015		11/21/2007	
									QC Type	SA		SA		SA		SA		SA		SA	
									Study ID	LTM		LTM		LTM		LTM		LTM		LTM	
									Sample Round	6		7		8		9		10		1	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value	Q	Value	Q	Value	Q	Value	Q	Value	Q	Value	Q
Inorganics																					
Copper	UG/L	0	0%	GA	200	0	0	70		25	U	1.9	U	1.9	U	1.9	U	1.8	U	20	U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		1.07	U	4	U	4	U	4	U	3.9	U	5	U

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
Q = Qualifier
U = compound was not detected
J = the reported value is an estimated concentration
SA = Field Sample
DU = Field Sample Duplicate

Appendix B
Complete Groundwater Monitoring Results for OB Grounds LTM
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

									Area	OB Grounds		OB Grounds		OB Grounds		OB Grounds
									Loc ID	MW23-2		MW23-2		MW23-2		MW23-2
									Matrix	GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER
									Sample ID	OBLM20010		OBLM20016		OBLM20017		OBLM20023		OBLM20030		OBLM20037
									Sample Date	2/25/2008		5/21/2008		5/21/2008		8/26/2008		8/3/2010		10/5/2011
									QC Type	SA		SA		DU		SA		SA		SA
									Study ID	LTM		LTM		LTM		LTM		LTM		LTM
									Sample Round	2		3		3		4		5		6
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value	Q	Value	Q	Value	Q	Value	Q	Value	Q	Value	Q
Inorganics																					
Copper	UG/L	0	0%	GA	200	0	0	70		20	U	20	U	20	U	20	U	20	U	25	U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		5	U	5	U	5	U	5	U	1.87	U	1.07	U

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
Q = Qualifier
U = compound was not detected
J = the reported value is an estimated concentration
SA = Field Sample
DU = Field Sample Duplicate

Appendix B
Complete Groundwater Monitoring Results for OB Grounds LTM
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

									Area	OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds	
									Loc ID	MW23-2		MW23-2		MW23-2		MW23-2		MW23-2		MW23-3	
									Matrix	GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	
									Sample ID	OBLM20044		OBLM20051		OBLM20058		OBLM20065		OBLM20066		OBLM20003	
									Sample Date	10/9/2012		12/11/2013		10/16/2014		10/21/2015		10/21/2015		11/21/2007	
									QC Type	SA		SA		SA		SA		DU		SA	
									Study ID	LTM		LTM		LTM		LTM		LTM		LTM	
									Sample Round	7		8		9		10		10		1	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value	Q	Value	Q	Value	Q	Value	Q	Value	Q	Value	Q
Inorganics																					
Copper	UG/L	0	0%	GA	200	0	0	70		1.9	U	1.9	U	1.9	U	1.8	U	1.8	U	20	U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		4	U	4	U	4	U	3.9	U	3.9	U	5	U

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
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Appendix B
Complete Groundwater Monitoring Results for OB Grounds LTM
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									Area	OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds	
									Loc ID	MW23-3		MW23-3		MW23-3		MW23-3		MW23-3		MW23-3	
									Matrix	GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	
									Sample ID	OBLM20004		OBLM20011		OBLM20018		OBLM20024		OBLM20031		OBLM20038	
									Sample Date	11/21/2007		2/25/2008		5/21/2008		8/26/2008		8/2/2010		10/4/2011	
									QC Type	DU		SA		SA		SA		SA		SA	
									Study ID	LTM		LTM		LTM		LTM		LTM		LTM	
									Sample Round	1		2		3		4		5		6	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value	Q	Value	Q	Value	Q	Value	Q	Value	Q	Value	Q
Inorganics																					
Copper	UG/L	0	0%	GA	200	0	0	70		20	U	20	U	20	U	20	U	20	U	25	U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		5	U	5	U	5	U	5	U	1.87	U	1.07	U

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
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Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
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									Area	OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds	
									Loc ID	MW23-3		MW23-3		MW23-3		MW23-3		MW23-3		MW23-4	
									Matrix	GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	
									Sample ID	OBLM20045		OBLM20046		OBLM20052		OBLM20059		OBLM20067		OBLM20005	
									Sample Date	10/8/2012		10/8/2012		12/10/2013		10/16/2014		10/20/2015		11/21/2007	
									QC Type	SA		DU		SA		SA		SA		SA	
									Study ID	LTM		LTM		LTM		LTM		LTM		LTM	
									Sample Round	7		7		8		9		10		1	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value	Q	Value	Q	Value	Q	Value	Q	Value	Q	Value	Q
Inorganics																					
Copper	UG/L	0	0%	GA	200	0	0	70		1.9	U	1.9	U	1.9	U	1.9	U	1.8	U	20	U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		4	U	4	U	4	U	4	U	3.9	U	5	U

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
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									Area	OB Grounds	OB Grounds	OB Grounds	OB Grounds	OB Grounds	OB Grounds
									Loc ID	MW23-4	MW23-4	MW23-4	MW23-4	MW23-4	MW23-4
									Matrix	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER
									Sample ID	OBLM20012	OBLM20019	OBLM20025	OBLM20026	OBLM20032	OBLM20039
									Sample Date	3/3/2008	5/21/2008	8/25/2008	8/25/2008	8/2/2010	10/5/2011
									QC Type	SA	SA	SA	DU	SA	SA
									Study ID	LTM	LTM	LTM	LTM	LTM	LTM
									Sample Round	2	3	4	4	5	6
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value Q	Value Q	Value Q	Value Q	Value Q	Value Q
Inorganics															
Copper	UG/L	0	0%	GA	200	0	0	70		20 U	20 U	20 U	20 U	20 U	0.63 U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		5.4	5 U	5 U	5 U	2.7 J	1.07 U

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
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									Area	OB Grounds	OB Grounds	OB Grounds	OB Grounds	OB Grounds	OB Grounds
									Loc ID	MW23-4	MW23-4	MW23-4	MW23-4	MW23-4	MW23-5
									Matrix	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER
									Sample ID	OBLM20047	OBLM20053	OBLM20054	OBLM20060	OBLM20068	OBLM20006
									Sample Date	10/8/2012	12/10/2013	12/10/2013	10/15/2014	10/20/2015	11/21/2007
									QC Type	SA	SA	DU	SA	SA	SA
									Study ID	LTM	LTM	LTM	LTM	LTM	LTM
									Sample Round	7	8	8	9	10	1
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value Q	Value Q	Value Q	Value Q	Value Q	Value Q
Inorganics															
Copper	UG/L	0	0%	GA	200	0	0	70		1.9 U	1.9 U	1.9 U	1.9 U	1.8 U	20 U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		4 U	4 U	4 U	4 U	3.9 U	5 U

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
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									Area	OB Grounds	OB Grounds	OB Grounds	OB Grounds	OB Grounds	OB Grounds
									Loc ID	MW23-5	MW23-5	MW23-5	MW23-5	MW23-5	MW23-5
									Matrix	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER
									Sample ID	OBLM20013	OBLM20020	OBLM20027	OBLM20033	OBLM20034	OBLM20040
									Sample Date	2/26/2008	5/21/2008	8/25/2008	8/2/2010	8/2/2010	10/4/2011
									QC Type	SA	SA	SA	SA	DU	SA
									Study ID	LTM	LTM	LTM	LTM	LTM	LTM
									Sample Round	2	3	4	5	5	6
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value Q	Value Q	Value Q	Value Q	Value Q	Value Q
Inorganics															
Copper	UG/L	0	0%	GA	200	0	0	70		20 U	20 U	20 U	1.62 U	20 U	25 U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		5 U	5 U	5 U	1.87 U	2.4 J	1.1 J

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
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Complete Groundwater Monitoring Results for OB Grounds LTM
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									Area	OB Grounds	OB Grounds	OB Grounds	OB Grounds	OB Grounds	OB Grounds
									Loc ID	MW23-5	MW23-5	MW23-5	MW23-5	MW23-5	MW23-6
									Matrix	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER
									Sample ID	OBLM20048	OBLM20055	OBLM20061	OBLM20062	OBLM20069	OBLM20007
									Sample Date	10/8/2012	12/10/2013	10/15/2014	10/15/2014	10/20/2015	11/28/2007
									QC Type	SA	SA	SA	DU	SA	SA
									Study ID	LTM	LTM	LTM	LTM	LTM	LTM
									Sample Round	7	8	9	9	10	1
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value Q	Value Q	Value Q	Value Q	Value Q	Value Q
Inorganics															
Copper	UG/L	0	0%	GA	200	0	0	70		1.9 U	1.9 U	1.9 U	1.9 U	1.8 U	20 U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		4 U	4 U	4 U	4 U	3.9 U	5 U

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
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Complete Groundwater Monitoring Results for OB Grounds LTM
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									Area	OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds		OB Grounds	
									Loc ID	MW23-6		MW23-6		MW23-6		MW23-6		MW23-6		MW23-6	
									Matrix	GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	
									Sample ID	OBLM20014		OBLM20021		OBLM20028		OBLM20035		OBLM20041		OBLM20042	
									Sample Date	2/26/2008		5/20/2008		8/26/2008		8/3/2010		10/5/2011		10/5/2011	
									QC Type	SA		SA		SA		SA		SA		DU	
									Study ID	LTM		LTM		LTM		LTM		LTM		LTM	
									Sample Round	2		3		4		5		6		6	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value	Q	Value	Q	Value	Q	Value	Q	Value	Q	Value	Q
Inorganics																					
Copper	UG/L	0	0%	GA	200	0	0	70		20	U	20	U	20	U	20	U	25	U	25	U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		5	U	5	U	5	U	3.6	J	1.2	J	1.5	J

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
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Complete Groundwater Monitoring Results for OB Grounds LTM
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									Area	OB Grounds		OB Grounds		OB Grounds		OB Grounds	
									Loc ID	MW23-6		MW23-6		MW23-6		MW23-6	
									Matrix	GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	
									Sample ID	OBLM20049		OBLM20056		OBLM20063		OBLM20070	
									Sample Date	10/8/2012		12/10/2013		10/16/2014		10/20/2015	
									QC Type	SA		SA		SA		SA	
									Study ID	LTM		LTM		LTM		LTM	
									Sample Round	7		8		9		10	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed		Value	Q	Value	Q	Value	Q	Value	Q
Inorganics																	
Copper	UG/L	0	0%	GA	200	0	0	70		1.9	U	1.9	U	1.9	U	1.8	U
Lead	UG/L	5.4	10%	MCL	15	0	7	70		4	U	4	U	4	U	3.9	U

Notes:
1. Copper action level is from NYSDEC Class GA Groundwater Standard (TOGS 1.1.1, June 1998).
2. Lead action level is from US EPA Maximum Contaminant Limit (MCL),
Source http://www.epa.gov/safewater/mcl.html#inorganic.html
3. Round 6, 7, 8, 9, and 10 samples were analyzed by SW846-6010C. Rounds 1 through 5 were analyzed using SW846-6010B.
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APPENDIX C

LABORATORY REPORTS

Laboratory Reports are provided on the electronic (CD) version of this report.

APPENDIX D

DATA VALIDATION REPORT

PROJECT NAME/NO. OB Grounds LTM Round 10
SDG: 680-118112-1
FRACTION: Metals (copper and lead)
LAB: Test America - Savannah
MEDIA: Groundwater

CRITERIA	Did Analyses Meet all criteria as specified in the SOPS?	If no, specify analysis IDs which do not meet criteria	Comments/Qualifying Actions	Qualifiers Added?
Data Completeness, Holding Times & Preservation	Yes		The cooler temperature was 3.6°C upon receipt by the laboratory. All samples were received in good condition based on the laboratory login report. Sample pH was below 2. Holding time met criteria.	No
Calibration	Yes		Calibrations available, taken every ten samples, and within recovery limits (90-110%) for metals. Initial calibration R2 >0.99.	No
Blanks (method blank, prep blank)	Yes		ICB, CCBs, and preparation blank did not contain lead or copper. No rinsate blank was collected for this SDG.	No
Interference Check Sample	Yes		Met requirements (80-120%) for Copper and Lead.	No
CRQL Standard	Yes		CRQL Check Standards performed and within QC limit of 70-130%R.	No
Laboratory Control Sample	Yes		LCS results within limits (i.e., 80-120%) for copper and lead.	No
Duplicates	Yes		Laboratory duplicate analysis was not conducted for this SDG. A field duplicate pair (OBLM20065 and OBLM20066) was collected for this SDG. Copper and lead were not detected.	No
Spike Sample Analysis	Yes		Spike analysis was conducted for OBLM20065 and the spike results were within 75%-125% limits.	No
ICP Serial Dilution	Yes		ICP serial dilution was conducted for OBLM20065. QC results were within criteria.	No
Detection Limits	Yes		IDL's available used as reporting limits. IDLs of copper and lead are less than CRDLs. No action was taken.	No
ICP Linear Range	Yes		All results within the ICP linear range.	No

APPENDIX E

REEDER CREEK INSPECTION PHOTOS (OCTOBER 2015)

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Reeder Creek Inspection Photos (October 2015)
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Photo #1 – Looking southeast (upstream) down Reeder Creek. Creek embankments are vegetated and organics (leaves) are visible floating on the water's surface.

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Reeder Creek Inspection Photos (October 2015)
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Photo #2 – Looking northwest (downstream). Vegetated embankments (east and west) on each side of Reeder Creek. Water levels in the creek are low, similar to the 2014 inspection.

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Reeder Creek Inspection Photos (October 2015)
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Photo #3 – Looking down at the western embankment of Reeder Creek. Folded over branches and brush in background of photo indicate past large flow event within Reeder Creek. Fractured shale pieces exposed in the foreground of the photo.

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Reeder Creek Inspection Photos (October 2015)
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Photo #4 – Looking northwest (downstream) at Reeder Creek. Fallen leaves and other organics are grouping together, decomposing and settling to the creek bottom. Creek bottom consists of decomposing organics and brown slime-like material similar to that observed during past inspection.

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Reeder Creek Inspection Photos (October 2015)
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Photo #5 – Looking west at the embankment on the OB Grounds side of Reeder Creek. The embankment is steeply sloped with vegetation and apparent deer tracks leading down to the water's edge.

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Photo #6 – Looking down at the eastern embankment of Reeder Creek. Downed intertwined branches and exposed soil on embankment indicate a high flow event within Reeder Creek. Fractured shale and organics visible on right side of photo.

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Reeder Creek Inspection Photos (October 2015)
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Photo #7 – Looking southwest at Reeder Creek's western embankment. Considerable scouring on the embankment exposing soil from high flow event. Bottom of creek surface covered in fractured shale pieces and organics on top of bedrock.

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Reeder Creek Inspection Photos (October 2015)
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Photo #8 - Looking northwest (downstream) beyond the bounds of the creek bottom excavation. Significant accumulation of leaves and other organics on right side of photo.

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Photo #9 – Looking east at the flat, grassy embankment adjacent to Reeder Creek. Most of the vegetation was matted down and decomposing, indicating a high flow event through Reeder Creek. The low water level can be seen in the background flowing from right to left in this photo.

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Reeder Creek Inspection Photos (October 2015)
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Photo #10 – Looking southeast (upstream) in Reeder Creek. Leaves and other organics are accumulating in the shallow, slow moving waters. Each embankment is well vegetated.

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Reeder Creek Inspection Photos (October 2015)
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Photo #11 – Looking southeast (upstream) at the area where the two beaver dams were observed in 2013 and 2014. Most of the branches used to compose the dams had been swept away, and water was flowing freely through the dam (water level not high enough to flow through dam on right side of photo.

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Photo #12 – Looking southeast (upstream) at the western dam first observed in 2013. A considerable amount of the branches had been swept away when comparing to photos taken during previous year's creek inspection. (See Photo #9 in 2014).

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Reeder Creek Inspection Photos (October 2015)
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Photo #13 – Looking northwest (downstream) at Reeder Creek. Eastern beaver dam observed in 2013 and 2014 has been swept away allowing water to flow freely through channel. (See photo #10 in 2014)

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Photo #14 – Looking northwest (downstream) at formerly standing western beaver dam. Since the water is not pooling behind the dams, the water level is not high enough to flow towards the western dam.

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Photo #15 – Looking southwest at the western creek embankment (OB grounds side) of Reeder Creek. Local erosion of the embankment is present; however, no evidence present of overland flow occurring or excessive deposition of sediment into the creek. The embankment is densely vegetated.

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Photo #16 – Looking southeast (upstream) down Reeder Creek. Each embankment is densely vegetated with no signs of overland flow depositing soils into Reeder Creek. Creek bottom consisted of fractured shale and organics.

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Photo #17 – Looking northwest (downstream) in Reeder Creek. Each side of the creek is well vegetated. Fractured shale and brown slime-like material observed along the creek bottom.

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Photo #18 – Looking northwest (downstream) in Reeder Creek. Each side of the creek is well vegetated. Fractured shale and brown slime-like material was observed along the creek bottom. Exposed fractured shale can be seen in the background of the photo.

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Photo #19 – Looking southeast (upstream) down Reeder. Fractured shale observed along the creek floor.

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Reeder Creek Inspection Photos (October 2015)
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Photo #20 – Looking northeast at creek embankment on the non-OB grounds side of Reeder Creek. Embankment is well vegetated with apparent deer trail leading down to the water's edge.

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Reeder Creek Inspection Photos (October 2015)
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Photo #21 - Looking southwest at creek embankment on the OB grounds side of Reeder Creek. Embankment is well vegetated with apparent deer trail leading down to the water's edge. No evidence of overland flow depositing sediment into the creek.

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Reeder Creek Inspection Photos (October 2015)
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Photo #22 – Looking northwest (downstream) down Reeder Creek. Fractured shale pieces and organics (leaves) visible along creek bottom. Both embankments are steep and well vegetated. Water levels up to 2 feet deep in background of photo.

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Reeder Creek Inspection Photos (October 2015)
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Photo #23 – Looking southeast (upstream) at Reeder Creek. Fallen tree is traversing Reeder Creek. Creek bottom made up of fractured shale and decaying organics.

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Reeder Creek Inspection Photos (October 2015)
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Photo #24 – Looking northeast at the embankment on the opposite side of the OB Grounds. The embankment is steep and well vegetated with an apparent deer trail leading down to the creek. No evidence of overland flow depositing sediments in the creek.

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Photo #25 – Looking southeast (upstream) down Reeder Creek. Exposed fractured shale pieces visible along the creek bed.

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Reeder Creek Inspection Photos (October 2015)
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Photo #26 – Looking northwest (downstream) down Reeder Creek. Exposed fractured shale visible on right side of photo. Each embankment is densely vegetated.

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Photo #27 – Looking southwest at the embankment on the OB Grounds side of Reeder Creek. Visible soil within the embankment appears to show signs of scouring, likely during high flow event. No evidence of overland flow transporting soils from the OB Grounds to Reeder Creek. Fractured shale visible in foreground of photo.

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Reeder Creek Inspection Photos (October 2015)
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Photo #28 – Looking down at the floor of Reeder Creek. The downed tree is slightly impeding the flow, causing the water to slow and organics to group together upstream of the log. Fracture shale pieces and organics are visible beneath the surface of the water.

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Reeder Creek Inspection Photos (October 2015)
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Photo #29 – Looking southeast (upstream) at Reeder Creek. Fractured shale and organics are visible along the creek floor. Each embankment is densely vegetated.

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OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #30 – Looking southwest at the steep embankment on the OB Grounds side of Reeder Creek. Visible soil within the embankment appears to show signs of scouring, likely during high flow event. No evidence of overland flow transporting soils from the OB Grounds to Reeder Creek.

Appendix E
Reeder Creek Inspection Photos (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #31 – Looking southeast (upstream) at Reeder Creek. Fracture shale pieces intermingled with decaying organics can be seen on the creek floor.

Appendix E
Reeder Creek Inspection Photos (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #32 – Looking southeast (upstream) at Reeder Creek. Creek bottom covered in fractured shale, brown slime, and decaying organics. Abundant organics observed in this portion of the creek.

Appendix E
Reeder Creek Inspection Photos (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #33 – Looking west at the Reeder Creek embankment on the side of the OB Grounds. Visible soil within embankment shows signs of scouring. The roots from vegetation are helping keep embankment soils in place.

Appendix E
Reeder Creek Inspection Photos (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #34 – Looking southeast (upstream) down Reeder Creek. Creek bottom composed of shale is coated in the brown slime and organics. Each embankment is densely vegetated.

Appendix E
Reeder Creek Inspection Photos (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #35 – Looking at the west embankment of Reeder Creek. There is no evidence of overland flow from the OB Grounds depositing soils into Reeder Creek.

Appendix E
Reeder Creek Inspection Photos (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #36 – Looking southeast (upstream) at Reeder Creek, beyond the limits of the creek sediment excavation. Exposed fractured shale no longer visible, and instead the creek floor is covered in thick layer of brown slime/organics.

Appendix E
Reeder Creek Inspection Photos (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #37 – View of sand bags in drainage ditch that leads down to Reeder Creek, located between MW23-1 and MW23-2. Flow through the drainage ditch during strong rain events has caused minor erosion around the perimeter of the sandbags. Erosion has created a new preferential flow path around the sand bags.

APPENDIX F

STATISTICAL ANALYSIS OF LTM RESULTS

Appendix F
Statistical Analysis of LTM Results
Data Distribution Report
Well MW23-4

UCL Statistics for Data Sets with Non-Detects

User Selected Options	
Date/Time of Computation	12/14/2015 12:38:08 PM
From File	lead.xls
Full Precision	OFF
Confidence Coefficient	95%
Number of Bootstrap Operations	2000

LEAD

General Statistics

Total Number of Observations	12	Number of Distinct Observations	6
Number of Detects	2	Number of Non-Detects	10
Number of Distinct Detects	2	Number of Distinct Non-Detects	4
Minimum Detect	2.7	Minimum Non-Detect	1.07
Maximum Detect	5.4	Maximum Non-Detect	5
Variance Detects	3.645	Percent Non-Detects	83.33%
Mean Detects	4.05	SD Detects	1.909
Median Detects	4.05	CV Detects	0.471
Skewness Detects	N/A	Kurtosis Detects	N/A
Mean of Logged Detects	1.34	SD of Logged Detects	0.49

Warning: Data set has only 2 Detected Values.

This is not enough to compute meaningful or reliable statistics and estimates.

Normal GOF Test on Detects Only

Not Enough Data to Perform GOF Test

Kaplan-Meier (KM) Statistics using Normal Critical Values and other Nonparametric UCLs

Mean	2.178	Standard Error of Mean	0.846
SD	1.246	95% KM (BCA) UCL	N/A
95% KM (t) UCL	3.697	95% KM (Percentile Bootstrap) UCL	N/A
95% KM (z) UCL	3.569	95% KM Bootstrap t UCL	N/A
90% KM Chebyshev UCL	4.715	95% KM Chebyshev UCL	5.865
97.5% KM Chebyshev UCL	7.46	99% KM Chebyshev UCL	10.59

Gamma GOF Tests on Detected Observations Only

Not Enough Data to Perform GOF Test

Gamma Statistics on Detected Data Only

k hat (MLE)	8.653	k star (bias corrected MLE)	N/A
Theta hat (MLE)	0.468	Theta star (bias corrected MLE)	N/A
nu hat (MLE)	34.61	nu star (bias corrected)	N/A
MLE Mean (bias corrected)	N/A	MLE Sd (bias corrected)	N/A

Appendix F
Statistical Analysis of LTM Results
Data Distribution Report
Well MW23-4

Gamma Kaplan-Meier (KM) Statistics

k hat (KM)	3.055	nu hat (KM)	73.32
		Adjusted Level of Significance (β)	0.029
Approximate Chi Square Value (73.32, α)	54.6	Adjusted Chi Square Value (73.32, β)	52.14
95% Gamma Approximate KM-UCL (use when n>=50)	2.925	95% Gamma Adjusted KM-UCL (use when n<50)	3.062

Lognormal GOF Test on Detected Observations Only

Not Enough Data to Perform GOF Test

Lognormal ROS Statistics Using Imputed Non-Detects

Mean in Original Scale	2.293	Mean in Log Scale	0.739
SD in Original Scale	1.148	SD in Log Scale	0.427
95% t UCL (assumes normality of ROS data)	2.888	95% Percentile Bootstrap UCL	2.837
95% BCA Bootstrap UCL	3.051	95% Bootstrap t UCL	3.253
95% H-UCL (Log ROS)	2.988		

DL/2 Statistics

DL/2 Normal		DL/2 Log-Transformed	
Mean in Original Scale	2.382	Mean in Log Scale	0.763
SD in Original Scale	1.106	SD in Log Scale	0.518
95% t UCL (Assumes normality)	2.955	95% H-Stat UCL	3.441

DL/2 is not a recommended method, provided for comparisons and historical reasons

Nonparametric Distribution Free UCL Statistics

Data do not follow a Discernible Distribution at 5% Significance Level

Suggested UCL to Use

95% KM (t) UCL	3.697	95% KM (% Bootstrap) UCL	N/A

Warning: One or more Recommended UCL(s) not available!

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

Appendix F
Statistical Analysis of LTM Results
Data Distribution Report
Well MW23-5

UCL Statistics for Data Sets with Non-Detects

User Selected Options	
Date/Time of Computation	12/14/2015 12:40:41 PM
From File	lead.xls
Full Precision	OFF
Confidence Coefficient	95%
Number of Bootstrap Operations	2000

LEAD

General Statistics

Total Number of Observations	12	Number of Distinct Observations	6
Number of Detects	2	Number of Non-Detects	10
Number of Distinct Detects	2	Number of Distinct Non-Detects	4
Minimum Detect	1.1	Minimum Non-Detect	1.87
Maximum Detect	2.4	Maximum Non-Detect	5
Variance Detects	0.845	Percent Non-Detects	83.33%
Mean Detects	1.75	SD Detects	0.919
Median Detects	1.75	CV Detects	0.525
Skewness Detects	N/A	Kurtosis Detects	N/A
Mean of Logged Detects	0.485	SD of Logged Detects	0.552

Warning: Data set has only 2 Detected Values.

This is not enough to compute meaningful or reliable statistics and estimates.

Normal GOF Test on Detects Only

Not Enough Data to Perform GOF Test

Kaplan-Meier (KM) Statistics using Normal Critical Values and other Nonparametric UCLs

Mean	1.533	Standard Error of Mean	0.5
SD	0.613	95% KM (BCA) UCL	N/A
95% KM (t) UCL	2.432	95% KM (Percentile Bootstrap) UCL	N/A
95% KM (z) UCL	2.356	95% KM Bootstrap t UCL	N/A
90% KM Chebyshev UCL	3.034	95% KM Chebyshev UCL	3.714
97.5% KM Chebyshev UCL	4.658	99% KM Chebyshev UCL	6.512

Gamma GOF Tests on Detected Observations Only

Not Enough Data to Perform GOF Test

Gamma Statistics on Detected Data Only

k hat (MLE)	6.899	k star (bias corrected MLE)	N/A
Theta hat (MLE)	0.254	Theta star (bias corrected MLE)	N/A
nu hat (MLE)	27.59	nu star (bias corrected)	N/A
MLE Mean (bias corrected)	N/A	MLE Sd (bias corrected)	N/A

Appendix F
Statistical Analysis of LTM Results
Data Distribution Report
Well MW23-5

Gamma Kaplan-Meier (KM) Statistics

k hat (KM)	6.26	nu hat (KM)	150.2
		Adjusted Level of Significance (β)	0.029
Approximate Chi Square Value (150.25, α)	122.9	Adjusted Chi Square Value (150.25, β)	119.1
95% Gamma Approximate KM-UCL (use when n>=50)	1.874	95% Gamma Adjusted KM-UCL (use when n<50)	1.934

Lognormal GOF Test on Detected Observations Only

Not Enough Data to Perform GOF Test

Lognormal ROS Statistics Using Imputed Non-Detects

Mean in Original Scale	1.49	Mean in Log Scale	0.341
SD in Original Scale	0.539	SD in Log Scale	0.353
95% t UCL (assumes normality of ROS data)	1.769	95% Percentile Bootstrap UCL	1.746
95% BCA Bootstrap UCL	1.786	95% Bootstrap t UCL	1.834
95% H-UCL (Log ROS)	1.846		

DL/2 Statistics

DL/2 Normal		DL/2 Log-Transformed	
Mean in Original Scale	2.032	Mean in Log Scale	0.667
SD in Original Scale	0.53	SD in Log Scale	0.325
95% t UCL (Assumes normality)	2.307	95% H-Stat UCL	2.487

DL/2 is not a recommended method, provided for comparisons and historical reasons

Nonparametric Distribution Free UCL Statistics

Data do not follow a Discernible Distribution at 5% Significance Level

Suggested UCL to Use

95% KM (t) UCL	2.432	95% KM (% Bootstrap) UCL	N/A

Warning: One or more Recommended UCL(s) not available!

Warning: Recommended UCL exceeds the maximum observation

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.

Recommendations are based upon data size, data distribution, and skewness.

These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).

However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

Appendix F
Statistical Analysis of LTM Results
Data Distribution Report
Well MW23-6

UCL Statistics for Data Sets with Non-Detects

User Selected Options	
Date/Time of Computation	12/14/2015 12:42:23 PM
From File	lead.xls
Full Precision	OFF
Confidence Coefficient	95%
Number of Bootstrap Operations	2000

LEAD

General Statistics

Total Number of Observations	11	Number of Distinct Observations	6
Number of Detects	3	Number of Non-Detects	8
Number of Distinct Detects	3	Number of Distinct Non-Detects	3
Minimum Detect	1.2	Minimum Non-Detect	3.9
Maximum Detect	3.6	Maximum Non-Detect	5
Variance Detects	1.71	Percent Non-Detects	72.73%
Mean Detects	2.1	SD Detects	1.308
Median Detects	1.5	CV Detects	0.623
Skewness Detects	1.63	Kurtosis Detects	N/A
Mean of Logged Detects	0.623	SD of Logged Detects	0.581

Warning: Data set has only 3 Detected Values.

This is not enough to compute meaningful or reliable statistics and estimates.

Normal GOF Test on Detects Only

Shapiro Wilk Test Statistic	0.842	**Shapiro Wilk GOF Test**	
5% Shapiro Wilk Critical Value	0.767	Detected Data appear Normal at 5% Significance Level	
Lilliefors Test Statistic	0.343	**Lilliefors GOF Test**	
5% Lilliefors Critical Value	0.512	Detected Data appear Normal at 5% Significance Level	

Detected Data appear Normal at 5% Significance Level

Kaplan-Meier (KM) Statistics using Normal Critical Values and other Nonparametric UCLs

Mean	2.1	Standard Error of Mean	0.755
SD	1.068	95% KM (BCA) UCL	N/A
95% KM (t) UCL	3.468	95% KM (Percentile Bootstrap) UCL	N/A
95% KM (z) UCL	3.342	95% KM Bootstrap t UCL	N/A
90% KM Chebyshev UCL	4.365	95% KM Chebyshev UCL	5.391
97.5% KM Chebyshev UCL	6.815	99% KM Chebyshev UCL	9.612

Gamma GOF Tests on Detected Observations Only

Not Enough Data to Perform GOF Test

Gamma Statistics on Detected Data Only

k hat (MLE)	4.36	k star (bias corrected MLE)	N/A
Theta hat (MLE)	0.482	Theta star (bias corrected MLE)	N/A
nu hat (MLE)	26.16	nu star (bias corrected)	N/A
MLE Mean (bias corrected)	N/A	MLE Sd (bias corrected)	N/A

Appendix F
Statistical Analysis of LTM Results
Data Distribution Report
Well MW23-6

Gamma Kaplan-Meier (KM) Statistics

k hat (KM)	3.868	nu hat (KM)	85.11
		Adjusted Level of Significance (β)	0.0278
Approximate Chi Square Value (85.11, α)	64.84	Adjusted Chi Square Value (85.11, β)	61.96
95% Gamma Approximate KM-UCL (use when n>=50)	2.756	95% Gamma Adjusted KM-UCL (use when n<50)	2.884

Lognormal GOF Test on Detected Observations Only

Shapiro Wilk Test Statistic	0.895	**Shapiro Wilk GOF Test**	
5% Shapiro Wilk Critical Value	0.767	Detected Data appear Lognormal at 5% Significance Level	
Lilliefors Test Statistic	0.313	**Lilliefors GOF Test**	
5% Lilliefors Critical Value	0.512	Detected Data appear Lognormal at 5% Significance Level	

Detected Data appear Lognormal at 5% Significance Level

Lognormal ROS Statistics Using Imputed Non-Detects

Mean in Original Scale	2.071	Mean in Log Scale	0.623
SD in Original Scale	1.006	SD in Log Scale	0.48
95% t UCL (assumes normality of ROS data)	2.621	95% Percentile Bootstrap UCL	2.559
95% BCA Bootstrap UCL	2.588	95% Bootstrap t UCL	2.732
95% H-UCL (Log ROS)	2.899		

UCLs using Lognormal Distribution and KM Estimates when Detected data are Lognormally Distributed

KM Mean (logged)	0.623	95% H-UCL (KM -Log)	2.877
KM SD (logged)	0.474	95% Critical H Value (KM-Log)	2.143
KM Standard Error of Mean (logged)	0.335		

DL/2 Statistics

DL/2 Normal		**DL/2 Log-Transformed**	
Mean in Original Scale	2.205	Mean in Log Scale	0.753
SD in Original Scale	0.632	SD in Log Scale	0.292
95% t UCL (Assumes normality)	2.55	95% H-Stat UCL	2.649

DL/2 is not a recommended method, provided for comparisons and historical reasons

Nonparametric Distribution Free UCL Statistics

Detected Data appear Normal Distributed at 5% Significance Level

Suggested UCL to Use

95% KM (t) UCL	3.468	95% KM (Percentile Bootstrap) UCL	N/A

Warning: One or more Recommended UCL(s) not available!

Note: Suggestions regarding the selection of a 95% UCL are provided to help the user to select the most appropriate 95% UCL.
Recommendations are based upon data size, data distribution, and skewness.
These recommendations are based upon the results of the simulation studies summarized in Singh, Maichle, and Lee (2006).
However, simulations results will not cover all Real World data sets; for additional insight the user may want to consult a statistician.

APPENDIX G

SOIL CAP INSPECTION PHOTO LOG (OCTOBER 2015)

Appendix G
Soil Cap Inspection Photo Log (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #01 – Grid Q8. No erosion or disturbances to the cap were observed. The grid is very well vegetated. View to the north.

Appendix G
Soil Cap Inspection Photo Log (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #02 – Grid R8. No erosion or disturbances to the cap were observed. The grid is very well vegetated. View to the east.

Appendix G
Soil Cap Inspection Photo Log (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #03 – Grid S8. No erosion or disturbances to the cap were observed. The grid is very well vegetated. View to the east.

Appendix G
Soil Cap Inspection Photo Log (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #04 – Grid P10. No erosion or disturbances to the cap were observed. The grid is well vegetated. View to the northwest.

Appendix G
Soil Cap Inspection Photo Log (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #05 – View to the west along the southern boundary of Grid I8. Patch along southern boundary of the grid (3-4 feet) is not as densely vegetated as the surrounding area. No change in conditions observed in previous years.

Appendix G
Soil Cap Inspection Photo Log (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #06 – Grid I6. No erosion or disturbances to the cap were observed. The grid is well vegetated. View to the northeast.

Appendix G
Soil Cap Inspection Photo Log (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #07 – Grid C2. No erosion or disturbances to the cap were observed. The grid is well vegetated. View to the northeast.

Appendix G
Soil Cap Inspection Photo Log (October 2015)
OB Grounds LTM 2015 Annual Report
Seneca Army Depot Activity

Photo #08 – Grid I6. Crushed shale used to fill and grade tire ruts in 2014 is still in place. No erosion or disturbances to the cap were observed. The grid is well vegetated. View to the north.

PARSONS

100 High Street • Boston, Massachusetts 02110-1713 • Phone (617) 946-9400 • Fax (617) 946-9777 • www.parsons.com

October 12, 2016

Ms. Amy Doss
U.S. Army Corps of Engineers
Engineering and Support Center, Huntsville
Attn: CEHNC-ED-CS-P
4820 University Square
Huntsville, Alabama 35816-1822

SUBJECT: **Draft 2016 Annual Long-Term Monitoring Report for the Fire Training and Demonstration Pad (SEAD-25) at Seneca Army Depot Activity in Romulus, NY; Contract W912DY-08-D-0003, Task Order 0015**

Dear Ms. Doss:

Parsons Federal (Parsons) is pleased to submit the Draft 2016 Annual Long-Term Monitoring Report for the Fire Training and Demonstration Pad (SEAD-25) at the Seneca Army Depot Activity (SEDA) in Romulus, New York. This work was performed in accordance with the Scope of Work for Task Order 0015 under Contract No. W912DY-08-D-0003.

This Report provides a review of long-term groundwater monitoring conducted during March 2016 and provides recommendations for future long-term monitoring at SEAD-25. This document also provides a review of the effectiveness of the remedy implemented at SEAD-25 in 2005.

Parsons appreciates the opportunity to provide you with this Report. Should you have any questions, please do not hesitate to call me at (617) 449-1405.

Sincerely,

Todd M. Heino, P.E.
Vice President

Enclosures

cc: R. Battaglia, USACE NY District
B. Frazier, USACE Huntsville
K. Hoddinott, USAPHC

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October 12, 2016

Mr. Julio Vazquez
USEPA Region II
Superfund Federal Facilities Section
290 Broadway, 18th Floor
New York, NY 10007-1866

Ms. Melissa Sweet
New York State Department of Environmental Conservation (NYSDEC)
Division of Environmental Remediation
625 Broadway, 12th Floor
Albany, NY 12233-7015

Mr. Mark Sergott
Bureau of Environmental Exposure Investigation
New York State Department of Health
Empire State Plaza Corning Tower, Room 1787
Albany, NY 12237

SUBJECT: **Draft 2016 Annual Long-Term Monitoring Report for the Fire Training and Demonstration Pad (SEAD-25) at Seneca Army Depot Activity in Romulus, NY; EPA Site ID# NY0213820830 and NY Site ID# 8-50-006**

Dear Mr. Vazquez/Ms. Sweet/Mr. Sergott:

Parsons Federal (Parsons) is pleased to submit the Draft 2016 Annual Long-Term Monitoring Report for the Fire Training and Demonstration Pad (SEAD-25) at the Seneca Army Depot Activity (SEDA) in Romulus, New York (EPA Site ID# NY0213820830 and NY Site ID# 8-50-006).

This Report provides a review of long-term groundwater monitoring conducted during March 2016 and provides recommendations for future long-term monitoring at SEAD-25. This document also provides a review of the effectiveness of the remedy implemented at SEAD-25 in 2005.

Parsons appreciates the opportunity to provide you with this Report. Should you have any questions, please do not hesitate to call me at (617) 449-1405.

Sincerely,

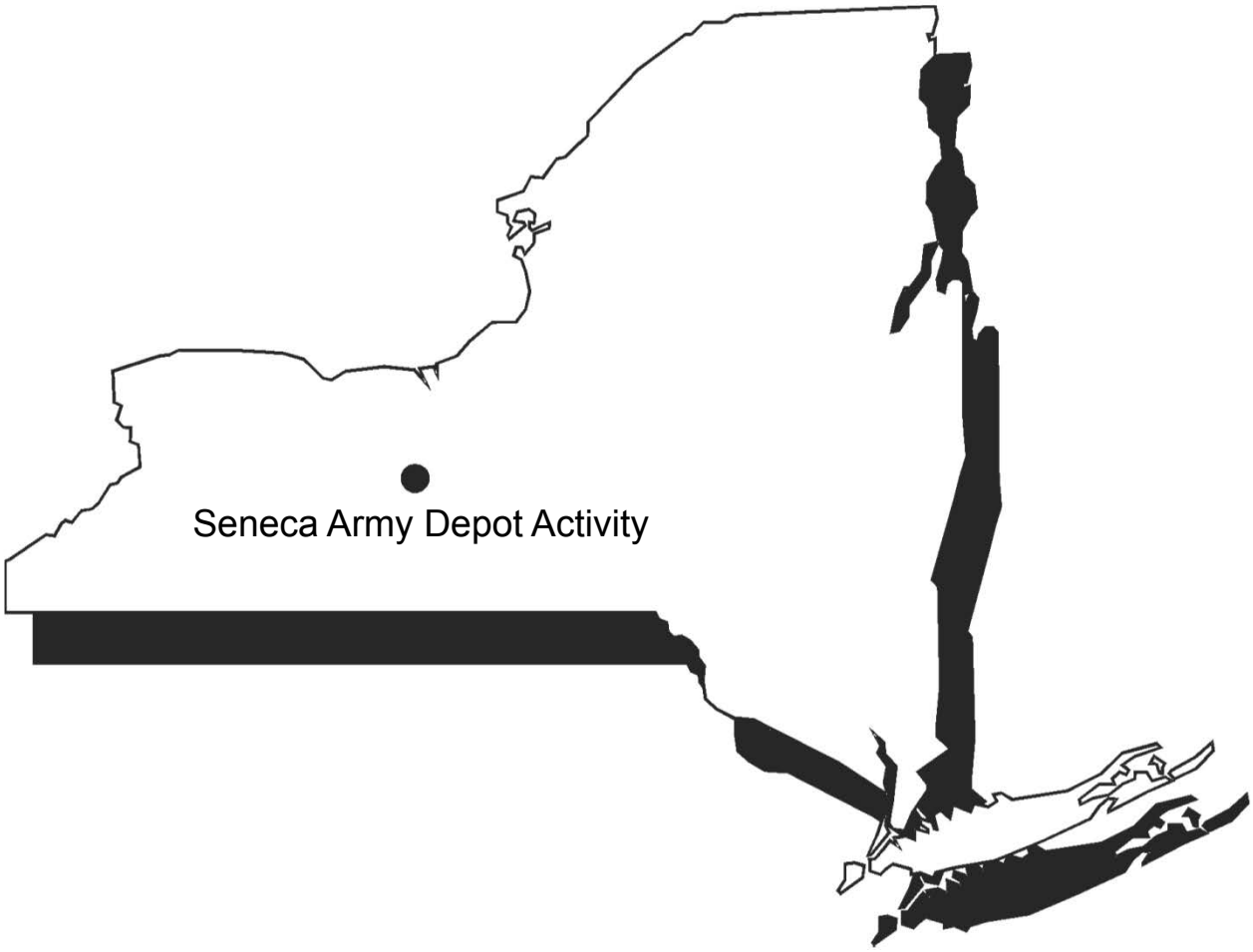
Todd Heino, P.E.
Vice President

Enclosures

cc: A. Doss, USACE Huntsville
B. Frazier, USACE Huntsville
R. Battaglia, USACE NY
K. Hoddinott, USAPHC

US Army, Engineering & Support Center
Huntsville, AL

Seneca Army Depot Activity
Romulus, NY



DRAFT

2016 LONG-TERM MONITORING ANNUAL REPORT

FOR THE FIRE TRAINING AND DEMONSTRATION PAD (SEAD-25)
SENECA ARMY DEPOT ACTIVITY

Contract No. W912DY-08-D-0003
Task Order No. 0015
EPA Site ID# NY0213820830
NY Site ID# 8-50-006

PARSONS

OCTOBER 2016

DRAFT

2016 LONG-TERM MONITORING ANNUAL REPORT

FOR THE FIRE TRAINING AND DEMONSTRATION PAD (SEAD-25)

SENECA ARMY DEPOT ACTIVITY, ROMULUS, NEW YORK

Prepared for:

U.S. ARMY, ENGINEERING & SUPPORT CENTER, HUNTSVILLE
4820 UNIVERSITY SQUARE
HUNTSVILLE, AL 35816
and
SENECA ARMY DEPOT ACTIVITY
ROMULUS, NEW YORK

Prepared by:

PARSONS
100 High Street
Boston, MA 02110

Contract Number W912DY-08-D-0003
Task Order No. 0015
EPA Site ID# NY0213820830
NY Site ID# 8-50-006

October 2016

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1.0 INTRODUCTION

This report provides a review of the 2016 (Event 13) long-term groundwater monitoring (LTM) sampling event conducted in March 2016 at the Fire Training and Demonstration Pad (SEAD-25) at the Seneca Army Depot Activity (SEDA or Depot) in Seneca County, New York. This document provides recommendations for future LTM and a review of the effectiveness of the remedy implemented at SEAD-25 in 2005. This report was issued by Parsons Federal (Parsons) on behalf of the U.S. Army (Army), Engineering and Support Center, Huntsville and the Seneca Army Depot Activity.

In accordance with the *Record of Decision* (ROD) *for the Fire Training and Demonstration Pad (SEAD-25) and the Fire Training Pit and Area (SEAD-26)* (Parsons, 2004) and the *Final Remedial Design Work Plan and Design Report* (RDR) *for the Fire Training and Demonstration Pad (SEAD-25) and the Fire Training Pit and Area (SEAD-26)* (Parsons, 2005a), a Remedial Action (RA) was completed in November 2005 for both area of concerns (AOCs), and the results of the actions were documented in the *Construction Completion Report for SEAD-25 and SEAD-26, Final* (CCR) (Parsons, 2006a). The SEAD-25 RA involved the removal of approximately 1,722 cubic yards (cy) of soil and sediment impacted by volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) at SEAD-25. With the approval of the Environmental Protection Agency (EPA) and the New York State Department of Environmental Conservation (NYSDEC), groundwater monitoring at SEAD-26 was terminated by the Army after the first year of sampling and analysis indicated that no COCs were present in the groundwater at concentrations above defined cleanup goals.

Long-term groundwater monitoring is being performed at SEAD-25 as part of the continuing post-closure monitoring and maintenance (PCMM) operations as described in the RDR. Groundwater monitoring was required at the AOC as a condition of the ROD since contaminant concentrations found in the groundwater at the AOCs prior to the RA exceeded applicable groundwater standards. Semi-annual groundwater monitoring of the ten monitoring wells (MW25-2, MW25-3, MW25-8, MW25-9, MW25-10, MW25-13, MW25-15, MW25-17, MW25-18, and MW25-19) located at SEAD-25 continued through 2013. The EPA and NYSDEC agreed, as recommended in the SEAD-25 *Fourth Long-Term Monitoring and Site Review Report* (Parsons, 2011c) and *Draft Final Five-Year Review Report* (Parsons, 2011d), to reduce the frequency of the semi-annual monitoring events to annual monitoring events. It was also agreed to reduce the number of wells to be monitored from ten to five since the down-gradient wells have shown no COCs during any of the post-RA sampling events. Beginning in 2014, the focus of the sampling effort is on wells MW25-2, MW25-3, MW25-9, MW25-10 and MW25-17 where historic information indicates that COCs of interest were detected.

Table 1 presents a summary of the historic LTM sampling and analysis events that were conducted at SEAD-25 since the completion of the RA activities. Thirteen (13) LTM sampling events, including the most current event completed in the first quarter of 2016 (2016Q1), were conducted at SEAD-25 since the completion of the RA at the site in late 2005. This *2016 Long-Term Monitoring Report* provides the details of LTM activities conducted during the annual LTM event in March 2016. This Report also provides an overall summary of the data collected at SEAD-25 since LTM began in late 2005.

2.0 SITE BACKGROUND

2.1 Site Description

The Seneca Army Depot is a 10,587-acre former military facility located in Seneca County in the towns of Romulus and Varick, New York, which was owned by the United States Government and operated by the Department of the Army between 1941 and 2000. The general location of the SEDA is shown on **Figure 1**. In 1999, SEDA's military mission was terminated and the installation was closed in 2000. Since 2000, the Army has assumed a caretaker role at the SEDA, pending the close-out of environmental investigations, studies, and remedial activities that are required at the former facility. As part of SEDA close-out activities, more than 8,250 acres of land within the former Depot was transferred to new owners for reuse.

The Seneca Army Depot is located between Seneca Lake and Cayuga Lake in Seneca County and is bordered by New York State Highway 96 on the east, New York State Highway 96A on the west, and sparsely populated farmland to the north and south. The Fire Training and Demonstration Pad (SEAD-25) is located in the east-central portion of SEDA. The site is bounded to the east by Administration Avenue, beyond which is undeveloped land covered by deciduous trees; to the south by Ordnance Drive beyond which is an open grassy field and a stand of coniferous trees; to the west by a drainage ditch running from the northeast to the southwest with grassland, brush and conifers between the site and the ditch; and, to the north by grassland and a former baseball field. A site map of the SEAD-25 area and its location within the SEDA is included as **Figure 2**. As situated, SEAD-25 sits a minimum of 1,350 feet away from the nearest SEDA boundary, which is located to the east of the AOC. A more detailed site map of SEAD-25 is provided as **Figure 3**. SEAD-25 was in use from the late 1960s to the late 1980s. The former pad was used for fire control training. During the 1980s, the pad was used twice for fire-fighting demonstrations, including one demonstration in 1982 or 1983, and one in 1987.

Site Hydrologic and Geologic Conditions

The hydrogeologic setting for SEAD-25 was previously described in detail in Section 3.1.6 of the *Final RI Report*[1] (RI Report) dated May 1998. A brief summary of hydrologic conditions described in the RI Report and historical groundwater conditions encountered during previous sampling events is presented below. Hydrologic conditions as observed during the 2016 LTM event are discussed in **Section 3.1** of this Report. Groundwater contours presented in the RI Report indicate that shallow groundwater flow below the pad is radial, with a stronger horizontal gradient to the south and west. The radial groundwater flow observed below the pad at SEAD-25 is believed to be a local phenomenon influenced by a bedrock topographic high located beneath the pad. The RI Report identified a west and southwest direction of groundwater flow in the deeper, competent shale bedrock.

The horizontal hydraulic gradients as presented in the RI Report ranged from 0.01 feet per foot (ft/ft) to 0.02 ft/ft in both the shallow saturated zone located in the till/weathered shale bedrock and in the deep saturated zone located in the competent shale bedrock. The hydraulic conductivities at SEAD-25 were found to range from 1.0×10^{-5} centimeters per second (cm/sec) to 3.4×10^{-3} cm/sec, with an average of 6.1×10^{-4} cm/sec in

[1] *Remedial Investigation Report for the Fire Training and Demonstration Pad (SEAD-25) and the Fire Training Pit and Area* (SEAD-26), Seneca Army Depot Activity, Parsons Engineering Science, Inc., May 1998

the shale/weathered bedrock. Both downward and upward vertical gradients were calculated for SEAD-25; the downward hydraulic gradients ranged from -0.04 ft/ft to -0.21 ft/ft, and upward hydraulic gradients ranged from 0.01 ft/ft to 0.07 ft/ft.

SEAD-25 is located very near a combined topographic and bedrock high within the east central portion of the former Depot. As such, all recharge to the local groundwater table comes from infiltration of storm-event precipitation which originates down through the surface into the underlying aquifer at, and in very close proximity to, the AOC. Infiltration rates are hindered because much of the storm-event precipitation is captured in neighboring drainage ditches and is conveyed to lower elevation areas within the Depot, which are down-gradient of the AOC's well recharge area.

The shallow overburden underlying SEAD-25 is thin, consisting of a till and fractured shale ranging from roughly 5 to 15 feet in thickness, which overlies competent shale bedrock. The monitoring wells sampled as part of SEAD-25 LTM effort are located in the shallow, overburden aquifer where the groundwater contamination was originally identified. As such, the combination of run-off and low infiltration or aquifer recharge periods that occur during extended dry or low water periods cause the overburden water table to thin to levels where samples cannot be collected from many of the wells and historically has not allowed a strict adherence to a semi-annual sampling schedule. This affects the collection of samples from one or more of the three source wells (MW25-2, MW25-3, and MW25-9). These wells are located closest to the former source area that was removed during the 2005 RA activities and historically have shown elevated levels of BTEX (i.e., benzene, toluene, ethyl benzene, and total xylenes) and chlorinated organic compound content.

2.2 Soil and Groundwater Impacts

As described in the RI Report (Parsons, 1998), the primary COCs historically observed at SEAD-25 included aromatic VOCs (benzene, toluene, ethyl benzene, and total xylenes) in soil and groundwater and lesser amounts of five chlorinated VOCs, including 1,1,1-trichloroethane, 1,1-dichloroethane (1,1-DCA), 1,2-dichloroethene (total) (1,2-DCE), chloroform, and trichloroethene (TCE), in groundwater. Vinyl chloride (VC), a degradation product of TCE and 1,2-DCE, was identified above its cleanup goal (2.0 micrograms per liter [µg /L]) at a concentration of 2.6 µg /L in MW25-2 during event 8 LTM and thus is included in the list of COCs at the site.

The pre-remedial action impacts from BTEX compounds occurred at three soil sample locations (SB25-3, SB25-4, and SB25-5) clustered together in the western half of the pad. The vertical impacts extended from the land surface to a depth of 4 to 6 feet below ground surface (bgs), which corresponds approximately to the top of competent bedrock (encountered at approximately 4.5 feet bgs during the RA). The highest concentrations of BTEX were detected at soil boring SB25-5, measuring 15,810 micrograms per kilogram (µg/kg), 151,500 µg/kg, and 10,200 µg/kg at depth intervals of 0-2 feet, 2-4 feet, and 4-6 feet bgs, respectively. Lower concentrations of BTEX were detected in the surface soil at sample locations SB25-3 (5,410 µg/kg) and at SB25-4 (2,900 µg/kg), respectively.

Impacts to soil located in the adjacent drainage swales at SEAD-25 were also noted and were mainly associated with SVOCs, pesticides, and heavy metals. The most significant impacts from SVOCs and

metals were found in the drainage swale northwest of the pad. In the ditch that runs along the west side of Administration Ave where it turns west along Ordnance Drive, the most significant SVOC impact was found in a single upgradient location. No COCs were identified in SEAD-25 surface water in concentrations that indicated remediation was required, and therefore remediation of surface water was not performed.

Based on the Final RI results, the primary groundwater impact was associated with two overlapping VOC plumes located in the overburden, both of which originated in the southwestern portion of the Fire Training and Demonstration Pad near the locations of the contaminated soil. Chlorinated ethenes and BTEX constituents were not detected in any of the bedrock wells at SEAD-25. The primary plume observed during the RI measured approximately 200 feet long and was composed of aromatic hydrocarbon compounds that are typically associated with gasoline (i.e., BTEX). The maximum concentration of total BTEX detected in the groundwater during the RI was 6,220 µg/L at well MW25-2. During the Expanded Site Investigation (ESI) (Parsons, December 1995), the maximum concentration of total chlorinated organics (96 µg/L) was also detected at well MW25-2.

2.3 Summary of the Remedial Action

The excavation of the BTEX-impacted soil at the SEAD-25 pad began on November 15, 2005 and was completed on December 1, 2005, with soil removal totaling approximately 961 cy. The depth of excavation extended to the top of the competent shale bedrock, or approximately 4.5 feet bgs. Ten confirmatory soil samples (plus one duplicate sample) were collected from the sidewalls of the excavation area and analyzed for VOCs and SVOCs. The analytical results of the confirmatory soil sample analyses achieved the site-specific cleanup goals, and the Army determined that soils at SEAD-25 did not require further action. The EPA and NYSDEC concurred with this determination that the excavation of the soil at the pad removed the source of groundwater contamination.

Excavation of the SVOC-impacted soil in the swale at SEAD-25 began on November 7, 2005 and was completed on November 8, 2005. The soil excavation extended to bedrock from the toe of slope on one bank to the toe of slope on the other bank, resulting in the removal and off-site disposal of approximately 761 cy of soil from SEAD-25. After the excavation, the swale bottom consisted of exposed competent bedrock, and since no native overburden soil remained in the swale, no confirmatory samples were collected or analyzed.

A total of approximately 1,722 cy (approximately 2,600 tons) of soil were excavated from the pad and the swale at SEAD-25 and disposed off-site at Ontario County Landfill. The pad excavation was backfilled with approximately 793 cy of on-site fill material and 168 cy of fill material obtained from an off-site source, and restored to the existing grade.

2.4 Natural Attenuation Process Evaluation

One of the purposes of long-term groundwater monitoring at SEAD-25 is to show that continued natural attenuation of the groundwater plume is occurring. This section gives a brief overview of the natural attenuation process and how the process can be evaluated. Numerous natural processes contribute to the reduction in dissolved phase contaminant concentrations over distance and time and are referred to as natural attenuation. These processes include sorption, dilution, dispersion, volatilization, and

biodegradation. Of these, biodegradation is of primary interest because this process destroys the contaminant, and because at many sites, it is the primary attenuation mechanism. The EPA's *Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Ground Water* (USEPA, 1998) can be used as guidance to determine if natural attenuation is occurring at SEAD-25.

Numerous laboratory and field studies have shown that many organic compounds are readily biodegraded via naturally occurring processes. Benzene and other petroleum hydrocarbons biodegrade readily under aerobic (oxygen-rich) conditions and have also been shown at multiple sites to biodegrade under anaerobic (oxygen-poor) conditions. Chlorinated ethenes biodegrade under anaerobic conditions through a process referred to as reductive dechlorination. Some chlorinated ethenes can also be biodegraded via direct aerobic oxidation (aerobic conditions).

Geochemical data including potential electron acceptors, biodegradation byproducts, and related analytes can be used as an indirect measure to show that organic compounds are biodegrading in saturated soil and groundwater. Depressed concentrations, when compared to background levels, of electron acceptors such as nitrate, oxygen, and sulfate that are used by microorganisms to facilitate the oxidation of VOCs within groundwater are geochemical indicators that VOCs are biodegrading. Similarly, elevated concentrations of biodegradation byproducts, such as iron II (Fe 2+), in groundwater are also geochemical indicators that compounds are biodegrading. Depressed oxidation/reduction potential (ORP) may also indicate the occurrence of biodegradation.

Biodegradation of chlorinated organics requires the presence of natural or anthropogenic carbon to create the conditions (anaerobic, low redox potential) necessary to stimulate reductive dechlorination of the more chlorinated solvents such as tetrachloroethene or perchloroethene (PCE) and TCE. Daughter products of these compounds (dichloroethene, or DCE; and VC) can be reductively dechlorinated under reducing conditions or directly oxidized under aerobic (oxidizing) conditions. Therefore, indicators of conditions appropriate for chlorinated biodegradation includes those parameters, such as methane, already identified for petroleum biodegradation and the presence of chlorinated daughter products and chloride. It should be noted, however, that the presence of road salt applied during the winter months may interfere with chloride data interpretation. The most common road salt is sodium chloride (NaCl), other commonly used road salt include calcium chloride (CaCl) and potassium chloride (KCl). Chloride ions are very soluble and mobile and can enter the groundwater by infiltration or surface water runoff.

Trends in natural attenuation parameters are more evident when higher concentrations of contaminants are present to naturally attenuate. At SEAD-25, trends in natural attenuation parameters are difficult to interpret since the contaminant concentrations are low, and have remained this way since the completion of the RA.

2.5 Well Decommissioning

The shallow saturated zone monitoring well MW25-11 and six deep saturated zone monitoring wells (MW25-4D, MW25-5D, MW25-7D, MW25-12D, MW25-14D, and MW25-16D) at SEAD-25 were removed in September 2010 as part of a SEDA-wide well decommissioning project; information pertinent to the well decommissioning project is provided in the *Final Well Decommissioning Report* (Parsons, 2013a). The location of decommissioned and existing SEAD-25 monitoring wells, including

latitude/longitude and northing/easting coordinates, and well elevation information, are provided in **Table 2**.

2.6 Land Use Control Inspection

SEAD-25 was inspected during the 2016 LTM event for compliance with the Land Use Control (LUC) restrictions that are in effect for AOCs located within the Planned Industrial/ Office Development (PID) and Warehouse Area at the former Depot. Land Use Controls for the PID/Warehouse Area implement and maintain requirements to:

- Prohibit the development and use of property for residential housing, elementary and secondary schools, childcare facilities, and playgrounds; and
- Prohibit access to or use of the groundwater, other than for monitoring purposes, until the applicable NYSDEC Class GA Groundwater Standards are met.

No residential housing units, elementary or secondary schools, childcare facilities or playgrounds were observed at SEAD-25. The 12 LTM groundwater monitoring wells were identified at SEAD-25 during the site visit. As discussed previously, many of the wells on the SEAD-25 site were decommissioned in September 2010.

3.0 LONG-TERM MONITORING RESULTS

3.1 2016 Sampling Event

The 2016 sampling event was completed at SEAD-25 between March 16 and March 17, 2016. Field forms documenting the collection of groundwater samples are provided in **Appendix A**. Groundwater laboratory analytical reports for this event are provided on a CD as **Appendix B**. Sampling procedures, sample handling and custody, holding times, and collection of field parameters were conducted in accordance with the *Final Sampling and Analysis Plan for Seneca Army Depot Activity* (SAP) (Parsons, 2005b).

Water level measurements were collected from the 12 monitoring wells at SEAD-25; however, as discussed above, only five wells (MW25-2, MW25-3, MW25-9, MW25-10 and MW25-17) were sampled. Groundwater samples were collected using low-flow sampling techniques and were analyzed for VOCs and natural attenuation parameters. A low-flow bladder pump was used to purge wells; following purging, samples were collected from each of the five wells for analysis of VOCs, sulfate, nitrate/nitrite, chloride, sodium, iron, methane, ethane, and ethene (MEE). Samples were submitted to TestAmerica in Savannah, Georgia. Analytes and analysis methods used are summarized below:

- VOCs - EPA SW846 Method 8260B
- MEE - RSK-175
- Nitrate and Nitrite - EPA Method 353.2
- Chloride - EPA Method 300.0
- Sulfate - EPA Method 300.0
- Iron - EPA SW846 Method 6010C
- Sodium -EPA SW846 Method 6010C

The TestAmerica Savannah, GA laboratory is certified by the Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) and the NELAC National Environmental Laboratory Accreditation Program (NELAP) for the above analyses/analytical methods for both potable and non-potable water.

Analytical results reported for the primary COCs (i.e., BTEX, and five chlorinated VOCs) and other detected VOCs were compared to New York State GA groundwater standards. Results of the other analyses conducted were used to assess if there is evidence that natural attenuation is occurring.

The following indicator and geochemical parameters were measured and recorded in the field:

- Sulfide
- pH
- Dissolved oxygen
- Conductivity
- Temperature
- ORP
- Turbidity

Indicator parameters including pH, ORP, conductivity, temperature, and turbidity of the groundwater were measured with a Horiba model U-52 water quality meter and dissolved oxygen (DO) content was measured with a YSI Inc. (YSI) model 85 DO Meter. Sulfide concentration was measured in the field using a Hach® colorimeter test at well locations. Indicator parameters were collected at all five wells (**Table 5**).

3.2 Groundwater Elevations

SEAD-25 Event 13 groundwater elevation data were recorded on March 16, 2016. Groundwater elevation data (events 10-13) and the historic post-2005 soil-removal action groundwater elevation range for the site are presented in **Table 3**. **Appendix C** provides groundwater elevations recorded from 2006 to 2016 and

groundwater elevation measurements performed between LTM sampling events. Groundwater elevation trends for SEAD-25 wells during the 13 LTM events performed from 2006 through 2016 are summarized on **Figure 4A** (Northern Profile) and **Figure 4B** (Southern Profile). Event 13 groundwater elevations ranged from 737.51 feet above mean sea level (amsl) in well MW25-13 to 742.98 feet amsl in well MW25-3. Groundwater elevations observed during this event are similar to those observed during the March 2015 (event 12) sampling event.

Groundwater contours were generated based on the groundwater elevation data collected on March 16, 2016 and are consistent with historic groundwater contour interpretation supporting the presence of a radial groundwater flow pattern beneath the pad (**Figure 5**). Contour interpretation indicates that shallow groundwater flow is radial, with the highest elevations located in the area of the former Fire Training and Demonstration Pad where soil removal was conducted in 2005.

3.3 Analytical Data Summary

3.3.1 2016 LTM Results

During the 2016 sampling event, six groundwater samples (including one duplicate sample from MW25-2) were collected for the analysis of VOCs. A summary of the primary COCs detected for event 13 are presented in **Table 4**, along with the applicable NYSDEC Class GA Groundwater Standards. The laboratory analysis reports are provided on a CD as **Appendix B**. A summary of the analytical results for each LTM event is provided in **Appendix D.** The data validation sheets are provided in **Appendix E;** there were no non-compliance issues reported.

During the 2016 sampling event, one VOC was detected in two samples. Benzene was detected at concentrations of 1.1 µg/L and 1.0 µg/L in the parent and duplicate samples collected from well MW25-2. Both concentrations were above the applicable groundwater cleanup standard (1 µg/L). No other VOCs were detected in any of the other sampled wells.

A summary of the range of concentrations for the primary COCs found during the SEAD-25 LTM monitoring event is presented below. At the exception of benzene in monitoring well MW25-2, results from the 2016 sampling event indicate that none of the other primary COCs exceeded applicable groundwater cleanup standards.

SEAD-25 2016 LTM Concentration Ranges Compared to NYS Class GA Groundwater Standards		
COCs	**SEAD-25 2016 LTM Concentration Range (µg/L)**	**NYSDEC GA Groundwater Standard (µg/L)**
Benzene *	1.1 – 1.0	1
Toluene *	ND	5
Ethylbenzene *	ND	5
Xylene (total) *	ND	5
Ortho Xylene	NA	5
Meta/Para Xylene	NA	5
1,1,1-Trichloroethane *	ND	5
1,1-Dichloroethane (DCA)*	ND	5
1,2-DCE (total) *	ND	5
Cis-1,2- DCE	ND	5
Trans-DCE	ND	5
Chloroform *	ND	7
Trichloroethene *	ND	5
Vinyl chloride	ND	2

Notes:
* = Primary COCs, signified with *, and other detected VOCs used to calculate total chlorinated organics with concentrations in excess of GA groundwater standards during annual events, are reported.
NA = Not Analyzed; ND = non-detect; J = estimated value

3.3.2 SEAD-25 LTM Analytical Summary

A summary of the historic groundwater sampling results for total BTEX and total chlorinated organics at SEAD-25 for the period from November 1995 (pre-RA) to March 2016 is presented on **Figure 6**. Total BTEX values were calculated using the following VOCs:

- benzene
- toluene
- ethyl benzene
- ortho xylene & meta/para xylene (if xylene total was not reported)
- xylene total (if meta/para and ortho xylenes were not reported)

Total chlorinated organics were calculated using the following VOCs:

- 1,1,1-trichloroethane
- 1,1-dichloroethane
- 1,2-dichloroethene total (if reported in lieu of cis- and trans-)
- cis-1,2-dichloroethene (if 1,2-dichloroethene total was not reported)
- trichloroethene
- chloroform
- vinyl chloride

3.3.2.1 BTEX Analytical Summary

Analytical results from LTM since 1995 indicate that BTEX compounds were only observed in the three source wells at SEAD-25 (i.e., MW25-2, MW25-3, and MW25-9). Generally, these data indicate that the pre-RA (1993-1996) groundwater concentrations of BTEX compounds decreased once the RA was completed in 2006. Since the RA was completed, BTEX contaminants identified at SEAD-25 predominantly were detected in source wells MW25-2 and MW25-9, and less frequently in source well MW25-3.

Total BTEX concentrations in well MW25-2 ranged from 115.6 J µg/L (event 7) to a minimum concentration of 0.64 J µg/L (event 12) (**Figures 6 and 7A**). Historically, benzene and ethyl benzene are the contaminants most frequently detected in MW25-2 and are the contaminants most frequently found at levels above their respective GA standards in this well. In the past two events (event 12 and 13), only benzene was detected in well MW25-2.

At MW25-3, after the completion of the RA, total BTEX concentrations have not exceeded 5.5 µg/L (event 11) and have been non-detect in eight of thirteen events (**Figures 6 and 7B**). The only BTEX compound to have exceeded its GA standard (1 µg/L) in this well is benzene - once in event 5 (1.7 µg/L) and once in event 11 (1.8 µg/L).

Total BTEX concentrations in groundwater collected from MW25-9 ranged from 124 µg/L (event 1) to non-detect (events 6, 12, and 13) (**Figures 6 and 7C**). Detections of BTEX compounds exceeded their respective GA standards in well MW25-9 five times (twice for benzene and once each for ethyl benzene, toluene, and total xylene). Four of these exceedances were observed during the first post-RA sampling event. Except for event 1, the only BTEX exceedance in well MW25-9 was benzene in event 4 (2.3 µg/L). No other BTEX components have exceeded their respective screening criteria in well MW25-9 except those detected during event 1.

3.3.2.2 Chlorinated COCs Analytical Summary

Analytical results from LTM since 1995 indicate that chlorinated organics were only observed in the three source wells at SEAD-25 (i.e., MW25-2, MW25-3, and MW25-9), with the exception of well MW25-10 with a concentration of 0.53 J µg/L (1,1,1-TCA) in event 2 and well MW25-19 where a concentration of 0.2 J µg/L (cis-1,2-DCE) was observed during event 3. The concentration of chlorinated COCs found in the groundwater at SEAD-25 decreased once the RA was completed and remained at non-detect to low aggregate part per billion (µg/L) concentrations in all wells until events 7 and 8 (**Figure 6).**

During events 7 and 8, chlorinated VOCs in MW25-2 were detected at concentrations higher than previous events (**Figure 8A**). Concentrations were found to decrease during event 9, but increased in events 10 and 11; however, the concentrations of individual chlorinated VOCs have remained below their applicable GA standards since event 9. The elevated concentrations in events 7, 8 and 11 correspond with periods of low groundwater elevation and are assumed to be elevated as a result of the limited saturated thickness of the groundwater table at these times (**Figure 4B**). Concentrations of chlorinated COCs in well MW25-2 ranged from 24.8 J µg/L (event 7) to non-detect (events 1, 2, 4, 12, and 13) (**Figures 6 and 8A**). In well MW25-2, individual chlorinated VOCs have not exceeded their applicable GA Standards since event 8.

Chlorinated COC concentrations in MW25-3 have been non-detect since the first RI (November 1995) (**Figures 6 and 8B**).

At well MW25-9, the total chlorinated COC concentration collected during event 1 was 5.44 µg/L; subsequent sampling events yielded non-detect values with the exception of event 12 (1.6 µg/L, TCE only) (**Figures 6 and 8C**). No individual chlorinated COCs have exceeded their applicable GA Standards in well MW25-9.

3.4 Data Trends and Natural Attenuation Evaluation

3.4.1 General Data Trends (VOCs)

There are two main lines of evidence to determine whether natural attenuation is occurring:

1. Reduction in contaminant concentrations; and
2. Indirect geochemical indicators to assess the groundwater's assimilative capacity.

The primary line of evidence, reduction in VOC concentrations, is the only direct measure of the attenuation of a plume. Since the completion of the remedial action at SEAD-25, benzene, ethyl benzene, toluene, and xylenes are the predominant aromatic VOCs detected in the groundwater. The detections of these VOCs are found exclusively at the three source wells (MW25-2, MW25-3, and MW25-9) with the majority of the benzene and ethyl benzene exceedances found in MW25-2. Over time, the BTEX concentrations have declined in these three wells with toluene and xylene concentrations generally non-detect in MW25-3 and MW25-9 since event 1 (**Appendix D**).

Total BTEX concentrations in the three source wells (MW25-2, MW25-3, and MW25-9) have decreased from pre-RA levels (**Figure 6** and **Figures 7A**, **7B**, and **7C**). At well MW25-2, BTEX concentrations fluctuate and have a moderate correlation with the saturated thickness at the time of sampling (**Figure 7A**). Elevated BTEX concentrations at MW25-2 are typically associated with lower groundwater levels. With two exceptions (events 5 and 11), BTEX concentrations at well MW25-3 are below their respective GA standards (**Figure 7B**). With the exception of events 1 and 4, BTEX concentrations are consistently below screening criteria in well MW25-9 (**Figure 7C**). Except for a limited exceedance of benzene (1.8 µg/L) in well MW25-3 in event 11, the BTEX concentrations of concern are restricted to well MW25-2.

Similarly, the concentrations of chlorinated COCs have decreased over time in the three source wells (**Figure 6** and **Figures 8A**, **8B**, and **8C**). The concentrations of chlorinated COCs in well MW25-2 are variable; however, only 1,2-DCE, cis-1,2-DCE, and VC have exceeded their individual cleanup standards during events 7 or 8. In wells MW25-3 and MW25-9, no chlorinated VOCs have exceeded their individual GA standards during LTM. Only MW25-2 exhibits concentrations of chlorinated VOCs that have exceeded their respective GA standards. The most recent exceedance of a chlorinated VOC in well MW25-2 was in event 8 in which all of the results were estimated (J flagged).

3.4.2 General Data Trends (Geochemical and Field Indicator Parameters)

Geochemical parameters (iron II, sodium, chloride, nitrate/nitrite, sulfate, and methane/ethane/ethene laboratory analysis, and field-measured DO, ORP and sulfide analysis) provide an indirect indication of the natural attenuation of the plume (**Table 5**). A review of historical field indicator data shows that no clear

trends of degradation are observed across SEAD-25; however, some parameters measured in the source wells suggest limited evidence for anaerobic biodegradation.

Methane was detected in the four of the five wells sampled during the 2016 sampling event at concentrations of 4.35 µg/L (MW25-2), 6.3 µg/L (MW25-3), 0.69 µg/L (MW25-9), and 0.29 J µg/L (MW25-17) (**Table 5**). During the 2016 sampling event, MW25-2 and MW25-3 yielded the highest detections of methane and no detections of chlorinated organics. Historical levels of methane measured in the source wells exhibit concentrations better than the suggested benchmark[2] of 0.5 mg/L or greater. The detection of methane in conjunction with BTEX COCs is interpreted to indicate that reductive dechlorination is occurring.

Concentrations of nitrate in the source wells are approximately equal to or better than the suggested benchmark[2] (< 1 mg/kg) for effective reductive dechlorination (**Table 5**). As discussed below, this value cannot be compared with background or upgradient concentrations to determine if an improving trend is found in the source area.

Parameters such as DO and ORP vary at each well location over time (**Table 5**). In the past, geochemical parameters have been conducive to reductive dechlorination (DO < 1 mg/kg and ORP < 50)[2]; however, a comparison in the data trend cannot be made as there is no upgradient well. At monitoring well MW25-2, the predominant source well, during events 2 through 11 and event 13, DO and ORP were measured at levels better than the suggested benchmark values for a likely reductive pathway (i.e., < 0.5 mg/L for DO and < 50 mV for ORP)[2]. Water level measurements from sampling event 12 indicated higher than normal groundwater conditions; the elevated levels of DO and ORP recorded in all of the wells during event 12 are assumed to be the result of an influx of fresh meltwater from the winter snowpack. During event 13 which happened at the same period of event 12, DO and ORP returned to historical values as the winter snowpack had already melted compared to event 12.

An assessment of other parameters (e.g., iron II, sodium, chloride) requires comparison to background concentrations or upgradient wells. Because of the radial groundwater flow pattern that exists at the site and the fact that the most contaminated wells are located near the central portion of the flow, determination of background conditions at SEAD-25 currently is not feasible. Overall, the review of the indicator parameters at well MW25-2 suggest that the VOCs are attenuating; indicator parameter results at the remaining monitoring wells are inconclusive due the historic lack of VOC contamination at these wells and the sporadic sampling frequency due to lack of water measured in the wells. Overall, the geochemical parameters for event 13 returned to historical levels observed prior to event 12 results. Although this makes interpreting data collected from this event difficult, the historical trends at the source wells continue to indicate an environment conducive to natural attenuation.

3.4.3 Data Trend Summary

Aromatic VOC concentrations in the three source wells (MW25-2, MW25-3, and MW25-9) generally indicate that the associated plume is attenuating (**Figures 9A, 9B**, and **9C**). Comparison of the pre- and post-RA groundwater concentrations at these wells demonstrate that the aromatic compound concentrations

[2] EPA (1998). Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater. September 1998.

have decreased significantly since the removal of the source area in late 2005. Prior to the remedial action the total aromatic COC concentration in well MW25-2 exceeded 5,000 µg/L. In wells MW25-3 and MW25-9, the total aromatic COC concentrations were approximately 200 µg/L.

Since the completion of the SEAD-25 RA, the total BTEX concentration in two of the three source wells has reached the GA standard. In the last six events, total BTEX concentrations in well MW25-2 have decreased to a maximum of 42 µg/L. BTEX components which exceed their GA Standard in well MW25-2 are limited to benzene and ethyl benzene. The historical pattern at MW25-2 indicates a fluctuation in the concentration that is thought to be related to the groundwater saturated thickness during the time of the sampling events. Further, MW25-2 is the only well at the site where BTEX COCs were detected in all of the consecutive LTM events until the 2015 event, suggesting that the overall groundwater impact has lessened and that BTEX COCs are not migrating. The detection of BTEX during the 2016 event is only due to the detection of benzene in MW25-2 and could not be interpreted as a regression in the historical trend observed.

Chlorinated VOCs contaminant distributions are similar to those observed for the BTEX VOCs. All of the noted exceedances of GA standards for the applicable chlorinated VOCs historically were observed at MW25-2.

Limited evidence for anaerobic biodegradation is supported by geochemical parameters such as low values of DO and negative values of ORP measured in the source wells. Methane and nitrate concentrations are generally better than their benchmark values indicating an environment where a reductive pathway is possible. Evidence that supports a subsurface environment that is conducive to natural attenuation in the area of the source wells suggests that COC concentrations in the area around well MW25-2 will decrease with time.

4.0 REMEDY EVALUATION

As discussed in Section 2.3, approximately 961 cy of VOC-impacted soil was removed from the location of the Fire Training and Demonstration Pad at SEAD-25 (**Figure 6)**. The soil was removed to eliminate the source of VOCs which could have contributed to further groundwater degradation in the area. Since 2006, long-term groundwater monitoring continues to be conducted at SEAD-25 to show that the soil removal remedy is effectively eliminating further VOC releases from the vicinity of the former pad and that natural attenuation of the VOC plumes at SEAD-25 continues to improve the groundwater quality.

Groundwater concentrations of BTEX and chlorinated organics have decreased by more than 99% since the soil removal due to the natural attenuation process and the removal of the source material during RA activities in 2005 (**Figures 7** and **8**). Soil removal therefore is determined to be an effective remedy at SEAD-25.

The remedy for SEAD-25 required the implementation and maintenance of LUCs. The LUC requirements are detailed in the Final Record of Decision for SEAD-25 and SEAD-26 (Parsons 2004), Addendum 1 in the *Land Use Control Remedial Design for SEAD 27, 66, 64A, Final* (2006) and are additionally covered under the area-wide LUCs Planned Industrial/Office or Warehousing Area ("PID Area") (Parsons, 2004; 2006b). The selected LUCs for SEAD-25 are as follows:

- Prevent residential housing, elementary and secondary schools, childcare facilities and playground activities; and
- Prevent access to and use of groundwater at SEAD-25, for purposes other than required monitoring, until NYS Class GA Groundwater Standards are met.

The areas of SEAD-25 were inspected to determine if the LUCs are being maintained. While performing the groundwater sampling, it was confirmed that at SEAD-25 no facilities, as described above, were constructed and no access to or use of groundwater, other than the collection of required LTM samples of groundwater, was evident.

5.0 LONG-TERM MONITORING CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

- The concentrations of BTEX in the groundwater at SEAD-25 have decreased by up to two orders of magnitude since 1994;
- With the exception of MW25-2, COCs were not detected above cleanup goals in four of the five wells sampled during the 2016 LTM event;
- VOC concentrations at SEAD-25 have generally attenuated to levels close to or below the applicable groundwater standards;
- The general trends of the field indicator parameters for most of the LTM wells provide inconclusive evidence due to the historic lack of VOC contamination at these wells and the lack of an upgradient or background well for comparison; however, typically low DO and negative ORP values at MW25-2 suggests an environment conducive to anaerobic degradation;
- COCs are limited in concentration and are not migrating outside the vicinity of MW25-2. In general, any remaining contamination is restricted to the area in the vicinity of MW25-2;
- Based on evaluation of available LTM data, the soil excavation remedy at SEAD-25 has been effective;
- The land and groundwater use restrictions imposed at SEAD-25 are maintained as part of both the approved ROD for SEAD-25 and the larger Planned Industrial/Office or Warehousing Area ("PID Area") (Parsons, 2004; 2006b). There are no signs of unauthorized use or access; and,
- Based on the information and discussion provided above, it appears that BTEX concentrations observed at MW25-2 fluctuate in correlation with changes in saturated thickness of the groundwater table, indicating that the increase is not due to the release of additional contaminants. The removal of the source area present at SEAD-25, and the verification that soils left at the site achieved cleanup objectives, supports the interpretation that a continuous release of contaminants at SEAD-25 is no longer occurring.

5.2 Recommendations

Based on the current area-wide LUC prohibiting the use of groundwater within the PID Area (which includes SEAD-25), the Army recommends concluding LTM at SEAD-25 because there is no planned future use of the groundwater. The wells will not be decommissioned at this time and sampling at these sites may take place in the future if the need arises (e.g., emerging contaminants, decisions during the 2021 5 Year Review). Annual LUC inspections will continue to insure that the groundwater is not accessed.

6.0 REFERENCES

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TABLES

Table 1
Summary of SEAD-25 Long-Term Monitoring Events
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

LTM Event Number	Sampling Event Designation[(1)]	Sampling Begin Date	Sampling End Date	Report Date	Report Type	Notes
Event 1	2006Q1	01/24/06	01/31/06	05/31/06	Technical Memo	One sample collected 04/12/06
Event 2	2006Q3	08/07/06	08/14/06	12/07/06 & 02/02/07	Technical Memo and Annual Report	Recommendation to terminate sampling at SEAD-26
Event 3	2007Q2	06/07/07	07/07/07	09/10/07	Technical Memo	
Event 4	2008Q1	03/03/08	03/04/08	04/18/08 & 06/18/08	Technical Memo and Annual Report	
Event 5	2009Q2	04/28/09	04/29/09	06/17/09	Technical Memo	
Event 6	2010Q1	01/11/10	01/14/10	01/21/11	Annual Report	Includes Event 5
Event 7	2010Q3	08/03/10	08/06/10	01/21/11	Technical Memo	
Event 8	2011Q1	02/07/11	02/10/11	05/26/11	Annual Report	Includes Event 7. Recommended to reduce semi-annual sampling to annual sampling and reduce number of wells to be sampled from 10 to 5 wells.
Event 9	2012Q1	02/28/12	03/02/12	04/10/13	Annual Report	
Event 10	2013Q2	05/06/13	05/09/13	04/11/14	Annual Report	
Event 11	2014Q2	06/17/14	06/18/14	02/25/15	Annual Report	Number of wells sampled reduced from 10 to 5 wells.
Event 12	2015Q1	03/16/15	03/18/15	08/19/15	Annual Report	
Event 13	2016Q1	03/16/16	03/17/15	06/30/16	Annual Report	

Notes:
(1) Event designation defined by year (XXXX) and quarter (QX) when samples were collected

Table 2
Monitoring Well Locations
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activitiy

Location ID	Northing (1)	Easting (1)	Loc_Elev (2)	Latitude (3)	Longitude (3)
MW25-1	998030.6639	751123.9323	740.3	42.73891679	-76.84050203
MW25-10	997966.2625	750999.2626	741.81	42.73873904	-76.84096538
MW25-11*	997865.7588	750955.8786	738.75	42.7384629	-76.84112574
MW25-12D*	997867.0397	750966.7103	738.89	42.7384665	-76.84108543
MW25-13	997864.8083	750869.3787	737.94	42.73845956	-76.84144772
MW25-14D*	997867.0994	750875.7165	738.23	42.7384659	-76.84142415
MW25-15	997972.6083	750764.5382	739.6	42.73875448	-76.84183921
MW25-16D*	997975.0098	750771.8704	739.75	42.73876113	-76.84181194
MW25-17	998188.4165	750964.1907	742.24	42.73934832	-76.84109846
MW25-18	998116.3641	751083.1527	743.05	42.73915161	-76.84065481
MW25-19	998136.6741	750763.1757	740.05	42.73920465	-76.84184615
MW25-2	998024.3007	750974.6108	743.76	42.73889808	-76.84105781
MW25-3	998079.4313	750926.4855	743.26	42.73904895	-76.84123758
MW25-4D*	998023.3883	750983.1189	743.81	42.73889565	-76.84102613
MW25-5D*	998081.3786	750938.3683	743.41	42.7390544	-76.84119337
MW25-6	998276.9972	751007.5574	742.24	42.73959174	-76.84093804
MW25-7D*	998279.0181	751016.2292	742.25	42.73959736	-76.84090578
MW25-8	998077.3072	750855.5452	741.36	42.73904253	-76.84150163
MW25-9	998004.1484	750898.1419	741.26	42.73884214	-76.84134223

Notes:

(1) Northing/Easting coordinates are based on New York State Plane NAD 83 coordinate system.

(2) Elevation measurements are based on New York State Plane NAD 83 coordinate system.

(3) Latitude and Longitude are in Universal Transverse Mercator (UTM) system and were obtained by converting the State Plane coordinates using U.S. Army Corps of Engineers Corpscon 6 software.

* = Indicates well was decommissioned in September 2010.
Bold location IDs denote the wells sampled in this event.

Table 3
SEAD-25 Groundwater Elevation Data
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Monitoring Well	Top of Riser Elevation (ft) [3]	Well Depth (ft)	Event 11 - June 17, 2014				Event 12 - March 16, 2015				Event 13 - March 16, 2016				LTM Rounds 1 through 13 Groundwater Elevation (ft) Max/Min Comparison and Range		
			Measured Well Depth (ft) [4]	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Measured Well Depth (ft) [4]	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Measured Well Depth (ft) [4]	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Maximum	Minimum	Range
MW25-1	743.00	7.77	7.73	1.16	6.57	736.43	7.71	2.88	4.83	738.17	7.55	2.37	5.18	737.82	738.17	736.41	1.76
MW25-2	746.36	11.31	11.26	4.35	6.91	739.45	11.25	7.37	3.88	742.48	11.05	7.21	3.84	742.52	742.52	738.54	3.98
MW25-3	746.34	9.58	9.80	2.01	7.79	738.55	9.80	6.71	3.09	743.25	9.55	6.19	3.36	742.98	743.25	737.58	5.67
MW25-6	744.44	14.27	14.30	6.37	7.93	736.51	14.27	11.34	2.93	741.51	13.70	10.80	2.90	741.54	741.54	735.89	5.65
MW25-8	742.46	5.47	5.42	0.38	5.04	737.42	5.44	3.93	1.51	740.95	5.20	3.70	1.50	740.96	740.96	737.30	3.66
MW25-9	742.36	5.42	5.40	0.45	4.95	737.41	5.40	4.07	1.33	741.03	5.20	3.70	1.50	740.86	741.03	737.35	3.68
MW25-10	743.01	6.20	6.39	0.26	6.13	736.88	6.38	5.00	1.38	741.63	6.15	3.93	2.22	740.79	741.63	736.88	4.75
MW25-13	739.64	5.70	5.48	0.33	5.15	734.49	5.47	2.81	2.66	736.98	5.25	3.12	2.13	737.51	737.51	734.46	3.05
MW25-15	741.00	7.20	7.20	0.23	6.97	734.03	7.20	5.23	1.97	739.03	6.95	4.42	2.53	738.47	739.03	734.03	5.00
MW25-17	743.94	11.60	11.26	4.48	6.78	737.16	11.24	9.52	1.72	742.22	10.72	8.92	1.80	742.14	742.22	736.49	5.73
MW25-18	744.35	11.00	11.18	4.28	6.90	737.45	11.16	7.84	3.32	741.03	11.00	7.30	3.70	740.65	744.20	737.13	7.07
MW25-19	741.95	12.10	12.00	3.54	8.46	733.49	12.01	7.87	4.14	737.81	11.80	8.90	2.90	739.05	739.05	732.92	6.13

Notes:

1. Groundwater levels were recorded in June 2014, March 2015 and March 2016.
2. Bedrock wells and well MW25-11 were decommissioned in September 2010 as part of the SEDA-wide Well Decommissioning Project.
3. Well MW25-3 total depth increased from 9 feet on 8/27/2008 to 9.58 feet on 4/29/2009. Groundwater levels after 8/27/2008 were adjusted to reflect the change in well total depth.
4. If well depths were not recorded during an event then the previously recorded well depth was used.

Table 4
SEAD-25 Primary COC Concentrations in Groundwater (Event 13)
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-10		MW25-17		MW25-2		MW25-2		MW25-3		MW25-9	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	
Sample ID									25LM20123		25LM20118		25LM20119		25LM20120		25LM20121		25LM20122	
Sample Date									3/17/2016		3/16/2016		3/17/2016		3/17/2016		3/17/2016		3/17/2016	
QC Type									SA		SA		SA		DU		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									13		13		13		13		13		13	
Filtered									Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
1,1,1-Trichloroethane	UG/L	0	0%	GA	5	0	0	6	0.37	U	0.37	U	0.37	U	0.37	U	0.37	U	0.37	U
1,1-Dichloroethane	UG/L	0	0%	GA	5	0	0	6	0.38	U	0.38	U	0.38	U	0.38	U	0.38	U	0.38	U
Chloroform	UG/L	0	0%	GA	7	0	0	6	0.5	U	0.5	U	0.5	U	0.5	U	0.5	U	0.5	U
Cis-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	6	0.41	U	0.41	U	0.41	U	0.41	U	0.41	U	0.41	U
Trichloroethene	UG/L	0	0%	GA	5	0	0	6	0.48	U	0.48	U	0.48	U	0.48	U	0.48	U	0.48	U
Vinyl chloride	UG/L	0	0%	GA	2	0	0	6	0.5	U	0.5	U	0.5	U	0.5	U	0.5	U	0.5	U
TOTAL Chlorinated Organics									**ND**		**ND**		**ND**		**ND**		**ND**		**ND**	
Benzene	UG/L	1.1	33%	GA	1	1	2	6	0.43	U	0.43	U	1		1.1		0.43	U	0.43	U
Ethyl benzene	UG/L	0	0%	GA	5	0	0	6	0.33	U	0.33	U	0.33	U	0.33	U	0.33	U	0.33	U
Toluene	UG/L	0	0%	GA	5	0	0	6	0.48	U	0.48	U	0.48	U	0.48	U	0.48	U	0.48	U
Total Xylenes	UG/L	0	0%	GA	5	0	0	6	0.23	U	0.23	U	0.23	U	0.23	U	0.23	U	0.23	U
TOTAL BTEX									**ND**		**ND**		**1**		**1.1**		**ND**		**ND**	

Notes:
1. Only primary COCs with site-specific cleanup goals are included.
2. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
3. Shading indicates concentration above cleanup goal.

J = the reported value is an estimated concentration
U = the compound was not detected
ND = Non-Detect

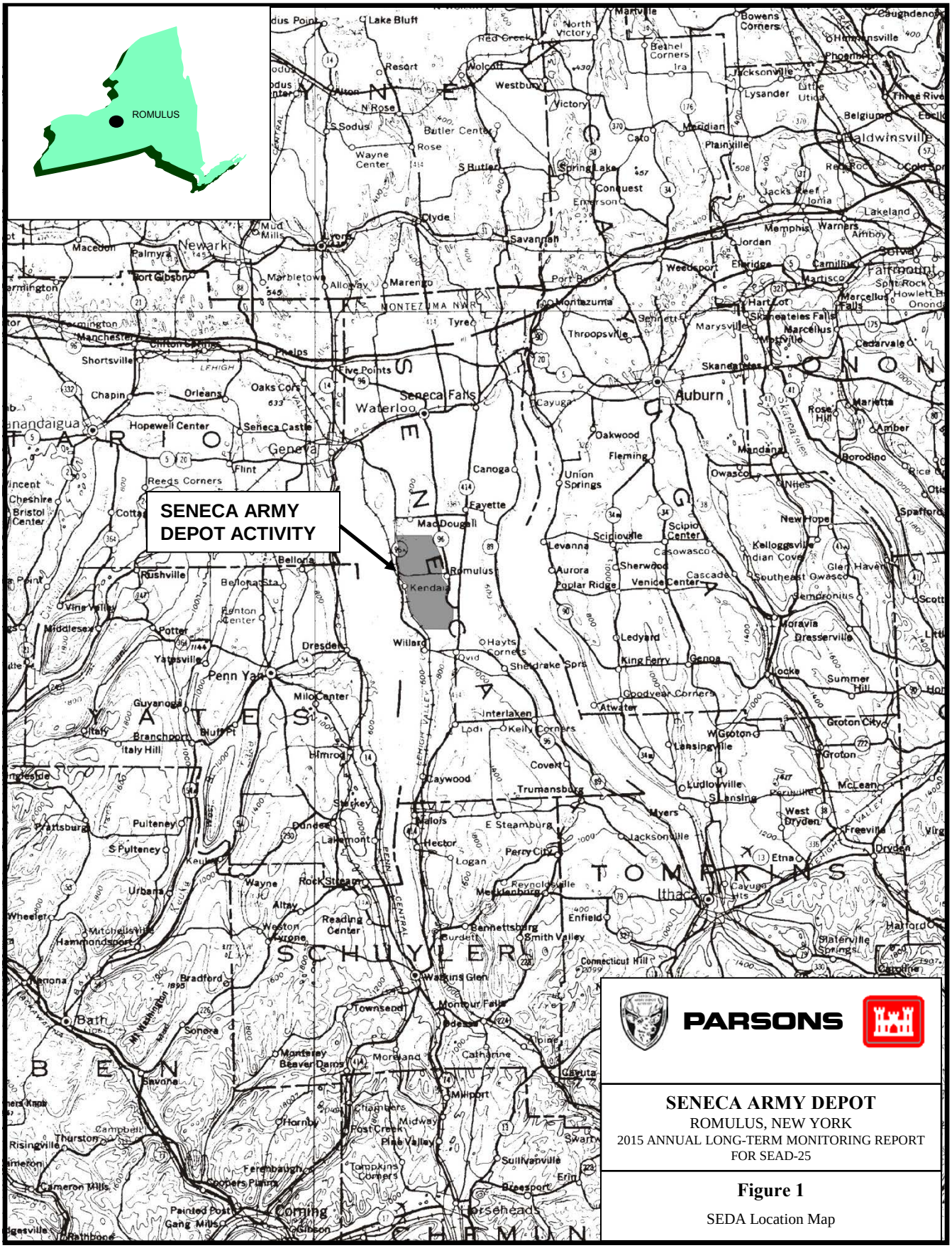
SA = Sample
DU = Duplicate

Table 5
Summary of SEAD-25 Geochemical Parameters
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Well ID	Date	Event	Dissolved Oxygen (mg/L)	ORP (mV)	Temperature (°C)	Turbidity (NTU)	pH (Std units)	Conductivity (S/m)	Iron (ug/L)	Sodium (ug/L)	Chloride (mg/L)	Nitrate (mg/L-N)	Nitrite (mg/L-N)	Sulfate (mg/L)	Ethane (ug/L)	Ethene (ug/L)	Methane (ug/L)	Sulfide (mg/L)
MW25-2	4/12/06 [1]	1	6.29	-11	10.5	16.1	7.17	0.551	2,510 J	4,730	6.5	0.05 U	0.05 U	39.6	2 U	2 U	80 J	0.01
	8/9/06 [1]	2	0.3	-82	26.55	2.3	6.93	0.562	667	5,600 J	2.2 J	0.05 U	0.05 U	32.1	10 U	10 U	35.5	0.15
	6/6/07	3	0.07	-92	12.4	11	7.11	0.454	2,600 J	6,000 J	4	0.5 J	0.5	22	0.24	4.2	170	--
	3/4/08	4	1.35	-60	3.2	2.78	7.15	0.64	711	3,460	0.2 U	0.305 J	0.305	31.1	1 U	1 U	3.2 J	0.01 U
	4/29/09 [1]	5	0.11	-115	8.1	0.9	6.84	0.702	15,050	7,100	2.2	0.05 U	0.01 U	79.2	1 U	1 U	66	0.04
	1/12/10 [1]	6	0.41	-151	6.3	1.06	7.25	0.573	2,655	7,800	2.8	0.199 J	0.007 UJ	64.6 J	0.16 U	0.17 U	21	0.16
	8/3/10 [1,4]	7	0.02	-230	21.2	3.4	6.79	1.09	1,660	10,300	2.9	0.013 UJ	--	42.8	0.16 U	0.17 U	125	--
	2/8/11 [1,6]	8	0.24	-148	5.08	0.6	6.98	0.806	13,100	10,200	5.8	0.0152 U	0.00321 U	45 J	0.58 U	0.69 U	45.5	--
	3/1/12	9	0.24	-106	5.3	5.38	6.79	0.681	3,780 J	9,320 J	0.9 J	0.0152 U	0.022 J	52 J	0.58 U	0.69 U	31 J	0.2
	5/8/2013 [1,6]	10	0.11	-350	8.4	3.11	7.20	0.907	8,750 J	13,000 J	1.75 J	0.0185 U	0.01 U	155 J	4 U	3 U	23.5	0.15
	6/18/14	11	0.68	-63	11.6	1.21	6.83	1.05	9,900	9,100	1.9	0.059	0.01 U	10	0.55 U	0.5 U	0.32 J	--
	3/18/15	12	8.84	44	3.3	1.48	7.57	0.411	340	2,500	0.74	1.3	0.01 U	24	0.55 U	0.5 U	4.2	0.00
	3/17/16 [1]	13	0.61	-17	5.1	3.67	7.74	0.669	345	3,550	0.735	0.36	0.01 J	36	0.55 U	0.5 U	4.35	0.03
MW25-3	1/31/06 [1]	1	1.19	79	4.3	2.2	7.1	0.49	81 J	12,150	2.2	0.05 U	0.05 U	39.85	2 U	2 U	2 U	0.04
	8/11/06	2	3.6	77.9	21.54	1.2	7.02	0.686	3,820	11,300 J	1.5 J	0.05 U	0.05 U	44.9	2 U	2 U	2 U	0.03
	3/4/08	4	0.87	124	3.5	2	7.15	0.675	107	5,540	2.66	0.098 J	0.01 UJ	100	1 U	1 U	0.34 J	0.01
	4/29/09	5	0.19	-102	7.9	0.35	7.03	0.627	1,570	9,000	3.3	0.05 U	0.01 U	122	1 U	1 U	13	0.42
	1/12/10	6	1.78	-63	4.9	3	6.51	0.741	702	7,370	2.8	0.05 UJ	0.007 UJ	182 J	0.16 U	0.17 U	0.14 U	0.04
	8/4/10 [4,5]	7	0	-124	20.6	2.37	6.84	1.26	--	--	--	--	--	--	0.16 U	0.17 U	12	--
	2/8/11 [6]	8	0.37	-85	4.5	3.31	6.99	0.851	463	7,990	3.2	0.057	0.00321 U	110 J	0.58 U	0.69 U	1.5 J	--
	2/29/12	9	0.1	-141	4.6	1.99	6.94	0.766	494 J	5,970	1.45 J	0.0152 U	0.0225 J	50 J	0.58 U	0.69 U	18 J	0.46
	5/9/13	10	0.25	-79	7.8	1.5	6.99	0.808	2,200	8,900	1 U	0.019 J	0.01 U	100	0.55 U	0.5 U	0.29 U	0.03
	6/18/14 [2,5]	11	--	--	--	--	--	--	--	--	--	--	--	--	0.55 U	0.5 U	11	--
	3/18/15 [1]	12	4.06	189	3.1	1.79	7.29	0.686	50 U	5,900	1.1	0.69	0.01 U	69	0.55 U	0.5 U	1.85	0.00
	3/17/16	13	0.95	-15	4.4	2.49	7.81	0.689	320	4,900	0.8	0.028 J	0.01 U	27	0.55 U	0.5 U	6.3	0.10
MW25-9	1/31/06	1	5.33	91	4.8	2.49	7.15	0.535	57 J	14,500	1.1	0.05 U	0.05 U	21.8	2 U	2 U	29	0.02
	8/9/06	2	5.22	62.5	23.11	3.38	7.15	0.718	12 U	16,400 J	0.99 J	0.1	0.05 U	25.3	2 U	2 U	2 U	0.45
	3/4/08	4	2.02	99	3.3	1.3	7.33	0.59	100 U	8,380	0.2 U	0.05 UJ	0.01 UJ	24.8	1 U	1 U	2.4 J	0.01 U
	4/29/09 [2,3]	5	--	--	--	--	--	--	9,440	26,000	2.7	0.05 U	0.01 U	39.7	1 U	1 U	3.5	0.12
	1/12/10 [3]	6	--	-72	3.62	2.8	6.73	0.427	916	16,500	0.5 U	0.05 UJ	0.007 UJ	35.3 J	0.16 U	0.17 U	0.14 U	0.01
	2/9/11 [2,3]	8	--	--	--	--	--	--	3,580	29,600	1.6 J	0.0152 U	0.00321 U	32 J	0.58 U	0.69 U	5.4 J	--
	2/29/12	9	1.77	-129	4.1	2.74	7.41	0.555	2,080 J	45,300	0.55 J	0.018 J	0.022 J	26 J	0.58 U	0.69 U	4 J	--
	5/7/13	10	0.16	-90	9.1	2.57	7.50	0.502	3,000	34,000	1 U	0.033 J	0.01 U	28	0.81 U	0.73 U	0.45 U	0.03
	6/18/14 [7]	11	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	3/17/15	12	10.97	192	2.2	4.81	7.73	0.423	92 J	14,000	2.3	0.85	0.013 J	25	0.55 U	0.5 U	0.8	0.02
	3/17/16	13	0.83	199	4.9	2.07	8.06	0.470	20 J	15,000	0.44 J	0.081	0.01 U	21	0.55 U	0.5 U	0.69	0.04
MW25-10	1/31/06	1	4.22	107	5	1.09	6.97	0.464	63 J	8,870	0.73	0.05 U	0.05 U	18.1	2 U	2 U	2 U	0.1
	8/9/06	2	4.23	138.8	21.56	195	6.56	0.701	358	6,530 J	0.71 J	0.05 U	0.05 U	18.4	2 U	2 U	2 U	0.28
	3/4/08	4	3.65	130	3.6	2.36	7.31	0.473	100 U	6,090	0.2 U	0.102 J	0.01 UJ	12.9	1 U	1 U	2 U	0.02
	1/13/10 [3]	6	--	230	5.6	3.3	7.19	0.396	508	6,420	2.1	0.05 UJ	0.007 UJ	27.1 J	0.21 U	0.22 U	0.14 U	0.09
	2/9/11 [2,5]	8	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	2/28/12	9	--	--	--	--	--	--	231 J	5,040	0.45 J	0.02 J	0.015 J	14 J	0.58 U	0.69 U	1.2 J	--
	5/7/2013 [6]	10	--	--	--	--	--	--	200 J	4,800 J	1 U	0.026 J	0.01 U	14 J	0.81 U	0.73 U	0.45 U	0.01
	6/18/14 [7]	11	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
	3/17/15	12	12.6	165	2.7	3.66	7.64	0.365	200	9,100	2.1	0.066	0.01 U	4.2	0.55 U	0.5 U	0.54 J	0.01
	3/17/16	13	8.36	175	5.7	1.13	7.99	0.389	25 J	5,000	0.73	0.082	0.01 U	8.5	0.55 U	0.5 U	0.29 U	0.01
MW25-17	1/31/06	1	8.46	68	6.3	3.4	7.69	0.462	46	4,240	0.7	0.05 U	0.05 U	17.2	2 U	2 U	2 U	0.01
	8/11/06	2	5.31	157	18.27	1.7	6.72	0.593	9 U	5,170 J	1.4 J	0.11	0.05 U	16.3	2 U	2 U	2 U	0.01 U
	6/7/07 [1]	3	0.31	134	13.2	12	7.2	0.418	440 J	8,500 J	3.6	3.44 J	0.73 J	18.5	0.23	1.3	6.55	0.06
	3/4/08 [1]	4	8.24	155	6	2.03	7.3	0.532	100 U	4,550	0.2 U	0.899 J	0.01 UJ	19.35	1 U	1 U	2 U	0.01
	4/28/09	5	7.45	192	7.2	1.2	7.31	0.379	160	4,700	0.2 U	0.05 U	0.01 U	17.3	1 U	1 U	2 U	0.01 U
	1/14/10	6	6.79	211	8.1	1.4	7.29	0.418	87 J	4,450	2.5	0.245 J	0.007 UJ	16.7 J	0.21 U	0.22 U	0.14 U	0.01 U
	8/5/10 [4]	7	4.1	61	17.6	2.45	7.25	0.584	56 J	5,650	5.3	0.484 J	--	21.7	0.16 U	0.17 U	0.14 U	0.01 U
	2/10/11	8	5.36	193	6.4	0	7.38	0.547	16 J	4,470	2.3	0.27	0.00321 U	16 J	0.58 U	0.69 U	0.98 J	0.01 U
	2/28/12	9	6.91	196	6.5	3.47	7.48	0.423	22 J	4,370	0.47 J	0.12	0.015 J	11 J	0.58 U	0.69 U	0.93 J	--
	5/8/13	10	6.52	73	7.4	2.48	7.76	0.558	50 U	5,500 J	1 U	0.19	0.01 U	18 J	0.81 U	0.73 U	0.45 U	0.01
	6/18/14 [1]	11	4.70	248	10.0	0.86	7.16	0.682	50 U	6,200	0.59	0.17	0.01 U	14	0.55 U	0.5 U	0.32 J	0.00
	3/17/15	12	5.59	224	5.0	1.65	7.51	0.520	50 U	5,200	1.2	0.24	0.01 U	16	0.55 U	0.5 U	0.96	0.01
	3/16/16	13	6.24	170	5.6	0.75	8.37	0.482	17 U	3,500	0.33 J	0.18	0.01 U	7.2	0.55 U	0.5 U	0.29 J	0.01

Notes:
- = geo parameter was not measured or sampled
1. Duplicate samples were averaged for available parameters.
2. Insufficient water volume to fill flow cell prior to sample collection.
3. Well was pumped dry and sampled the following day after recharge.
4. Lab analyzed for combined Nitrate/Nitrite Nitrogen.
5. Insufficient water to fill all the sample bottles; VOCs were collected and if additional water remained MEE was collected.
6. Well ran dry during sampling, allowed well to recharge overnight, and remaining samples were collected the next day.
7. Well was not sampled due to insuffcient water volume.

FIGURES



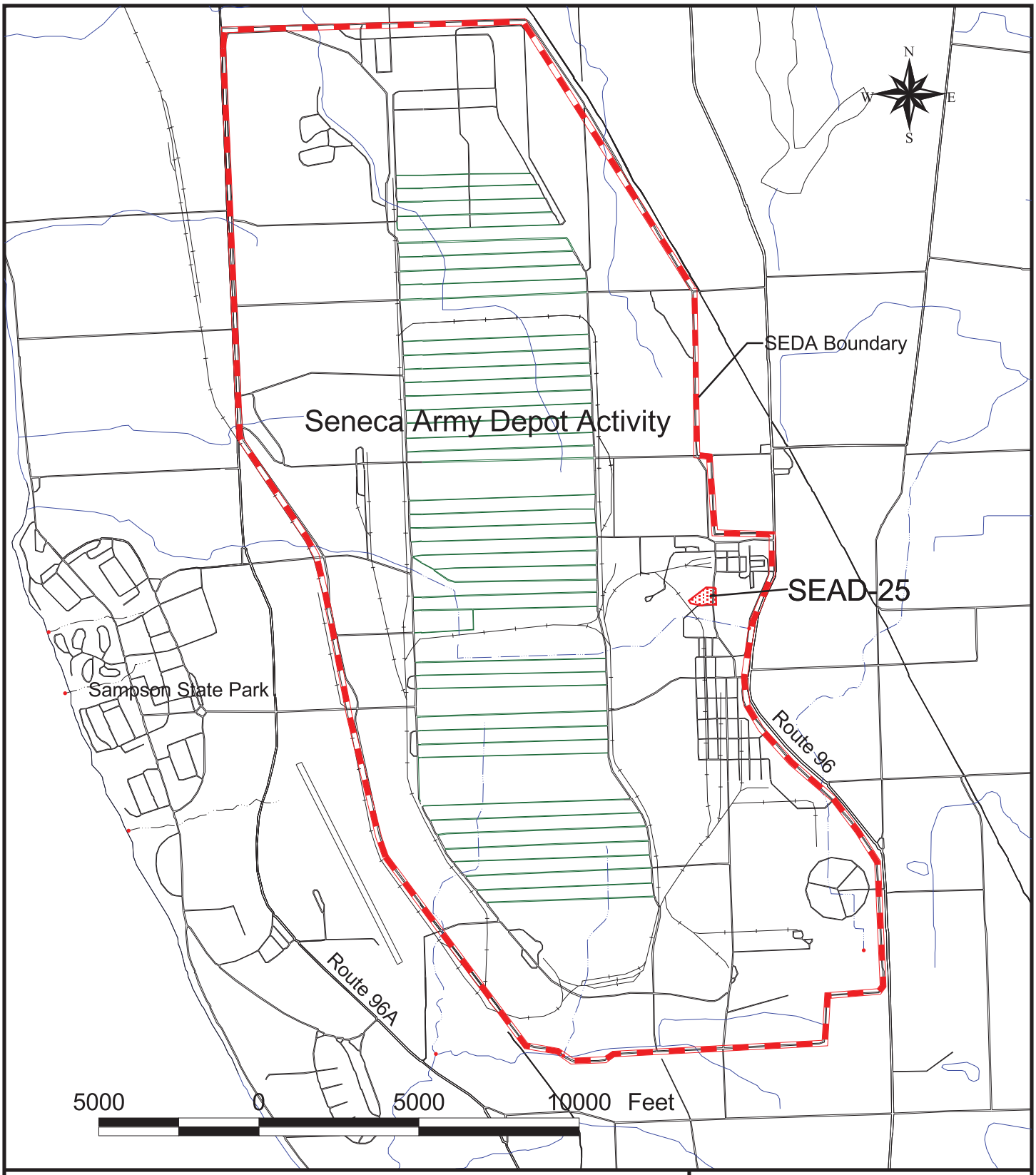
SENECA ARMY DEPOT

ROMULUS, NEW YORK

2015 ANNUAL LONG-TERM MONITORING REPORT FOR SEAD-25

Figure 1

SEDA Location Map



Approximate Boundary and extent of SEAD-25

Approximate Boundary of SEDA Site

PARSONS

CLIENT / PROJECT TITLE

SENECA ARMY DEPOT

ROMULUS, NEW YORK

2016 ANNUAL LONG-TERM MONITORING REPORT FOR SEAD-25

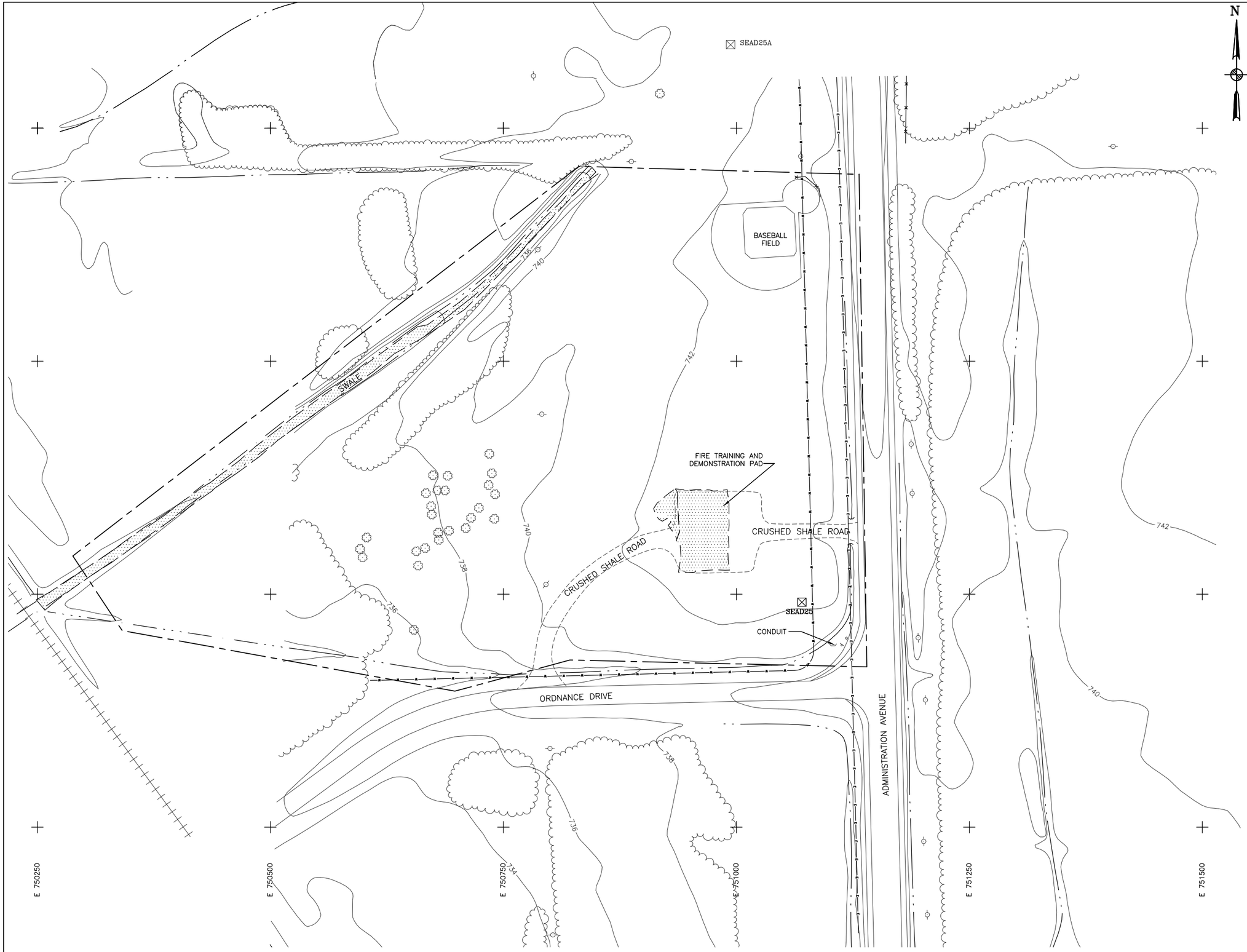
DEPT: ENVIRONMENTAL REMEDIATION

Figure 2

SEDA Site Map and AOC Location

DATE JUNE 2016

o:\seneca\sead2526\2526prap.apr\Annual Report Location



LEGEND

- DRAINAGE DITCH
- FENCE
- UNPAVED ROAD
- SEAD 25 BOUNDARY
- BRUSH LINE
- RAILROAD
- 760 — GROUND SURFACE ELEVATION CONTOUR
- UNDERGROUND ELECTRIC UTILITY LINE
- UNDERGROUND WATER UTILITY LINE
- ROAD SIGN
- OVERHEAD UTILITY POLE
- HYDRANT
- MANHOLE
- UTILITY BOX
- DECIDOUS TREE
- COORD. GRID (250' GRID)
- POLE
- SEAD-25 — SURVEY MONUMENT
- NOV/DEC 2005 REMEDIATED AREAS

NOTES:

1. TOPOGRAPHY BASED ON AERIAL SURVEY BY:

   LOCKWOOD SURVEY
   36 KARLAN DRIVE
   ROCHESTER NEW YORK

2. HORIZONTAL DATUM IS BASED ON NAD83 PER SENECA ARMY DEPOT SEAD 25A MONUMENTS SURVEY CONTROL COORDINATES DATED 1994.

3. VERTICAL DATUM IS BASED ON NAD88.

50 0 50 100
(feet)

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT
ROMULUS, NEW YORK
2016 ANNUAL LONG-TERM MONITORING REPORT FOR SEAD-25

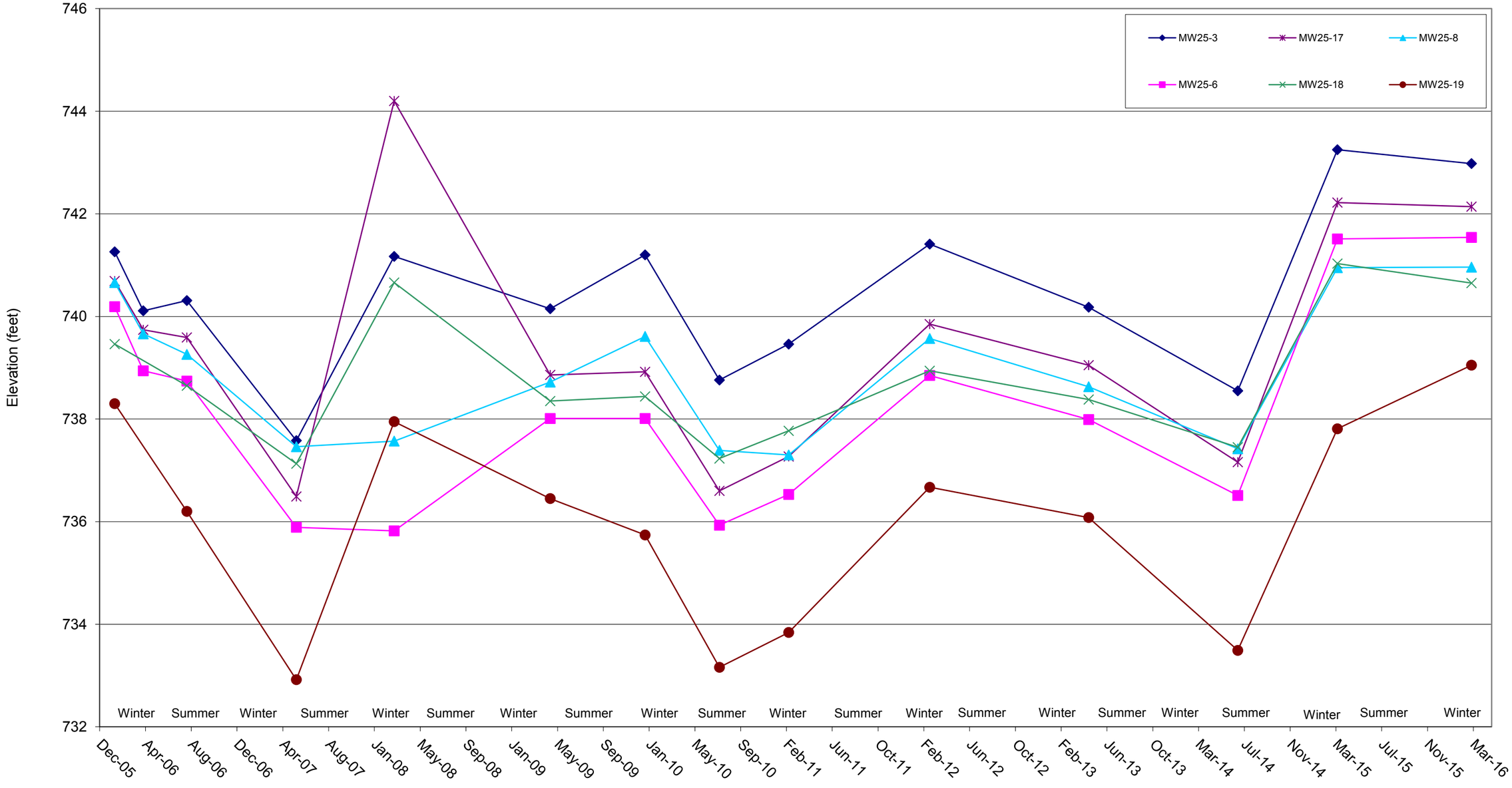
DEPT.	Dwg. No.
ENVIRONMENTAL ENGINEERING	

FIGURE 3
SEAD-25 SITE PLAN

SCALE	DATE	REV
AS SHOWN	June 2016	

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\SEAD 25\ANNUAL RPT 6 2013\DRAFT\FIGURES\FIGURE FILES-ACAD ETC\, DATE: 03/11/2014 06:12:58AM, P0042241

Figure 4A
SEAD-25 Groundwater Elevations - Northern Profile
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity



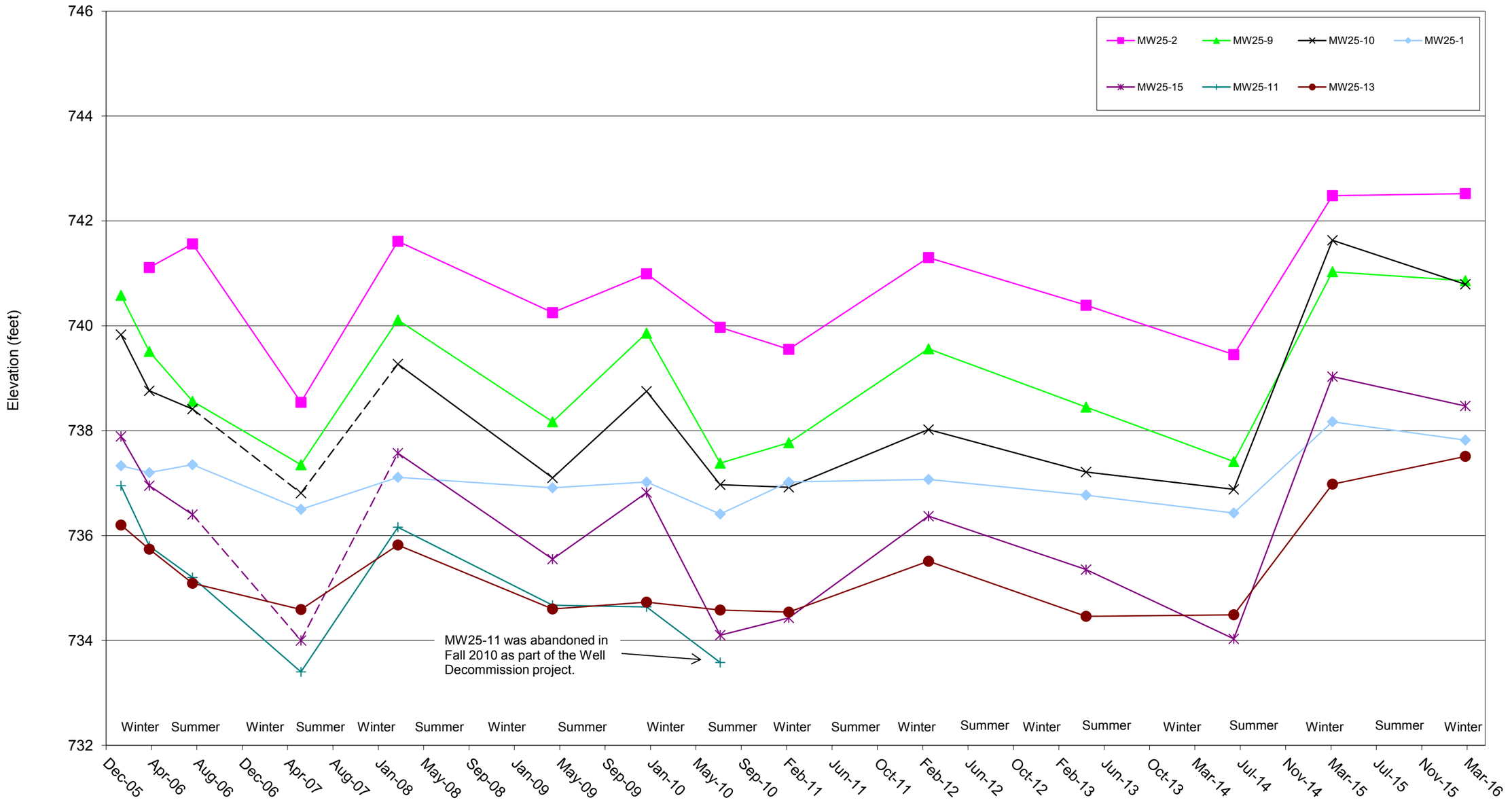
Notes:

1) Groundwater elevation was measured on the following dates: January 24, 2006; April 4, 2006; August 9, 2006; June 4, 2007; February 26, 2008; April 27, 2009; January 11, 2010; August 2, 2010; February 27, 2012; May 6, 2013; June 17, 2014; March 16, 2015; and March 16, 2016.

2) MW25-18 and MW25-19 groundwater elevations were not measured on April 4, 2006.

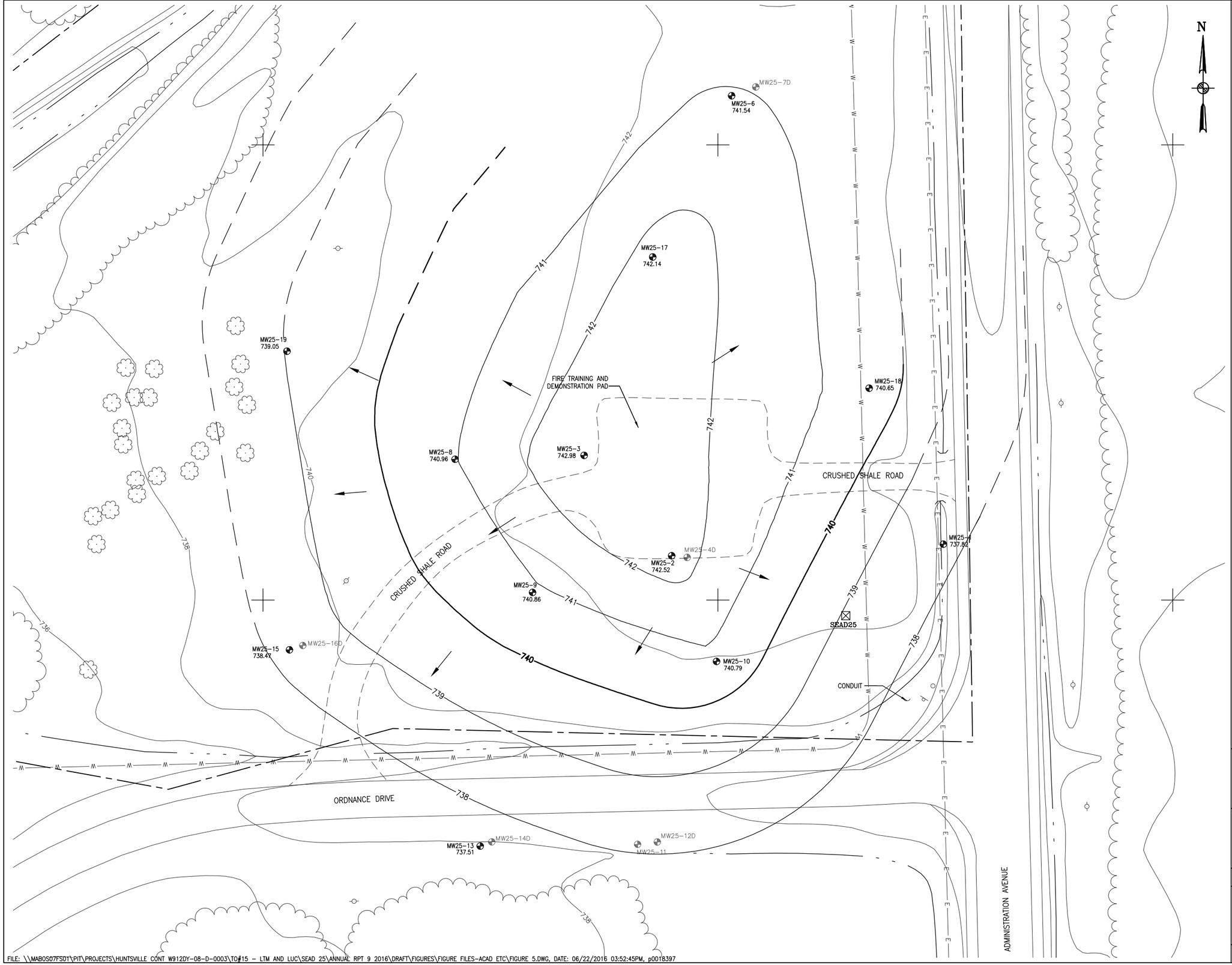
P:\PIT\Projects\Huntsville Cont W912DY-08-D-0003\TO#15 - LTM and LUC\SEAD 25\Annual Rpt 9 2016\Draft\Figures\Figure 4A-4B - GW Elev Trends_2016.xls\4A_S-25 GW Elev North Profile

Figure 4B
SEAD-25 Groundwater Elevations - Southern Profile
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity



Notes:

1) Groundwater elevation was measured on the following dates: January 24, 2006; April 4, 2006; August 9, 2006; June 4, 2007; February 26, 2008; April 27, 2009: January 11, 2010; August 2, 2010; February 7, 2011; February 27, 2012; May 6, 2013; June 17, 2014; March 16, 2015; and March 16, 2016.



LEGEND

- DRAINAGE DITCH
- FENCE
- UNPAVED ROAD
- SEAD 25 BOUNDARY
- BRUSH LINE
- RAILROAD
- 760 — GROUND SURFACE ELEVATION CONTOUR
- UNDERGROUND ELECTRIC UTILITY LINE
- UNDERGROUND WATER UTILITY LINE
- ROAD SIGN
- OVERHEAD UTILITY POLE
- HYDRANT
- MANHOLE
- UTILITY BOX
- DECIDOUS TREE
- COORD. GRID (250' GRID)
- POLE
- SEAD-25 — SURVEY MONUMENT
- MW25-19 740.1 — MONITORING WELL LOCATION & ELEVATION OF WATER TABLE
- MW25-19 — FORMER MONITORING WELL LOCATION
- 736 — GROUNDWATER CONTOUR (DASHED WHERE INFERRED)
- INDICATES PREDOMINANT FLOW DIRECTION

NOTE:

FORMER MONITORING WELLS WERE REMOVED IN SEPTEMBER 2010 AS PART OF THE WELL DECOMMISSIONING PROJECT.

25 0 25 50
(feet)

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT
ROMULUS, NEW YORK
2016 ANNUAL LONG-TERM MONITORING REPORT FOR SEAD-25

DEPT. ENVIRONMENTAL ENGINEERING | Dwg. No.

FIGURE 5
SEAD-25 GROUNDWATER CONTOURS
TILL/WEATHERED SHALE SATURATED ZONE
JUNE 2016

SCALE AS SHOWN | DATE JUNE 2016 | REV

FILE: \\MABOS07FS01\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\SEAD 25\ANNUAL RPT 9 2016\DRAFT\FIGURES\FIGURE FILES-ACAD ETC\FIGURE 5.DWG, DATE: 06/22/2016 03:52:45PM, p0018397

N

SEAD25A

8500

8250

8000

7750

E 750250

E 750500

E 750750

E 751000

E 751250

E 751500

50 0 50 100
(feet)

DATE	BTEX	TOT. CHLOR. ORGS.
MAR. 16	NA	NA
MAR. 15	NA	NA
JUN. 14	NA	NA
MAY 13	ND	ND
FEB. 12	ND	ND
FEB. 11	ND	ND
AUG. 10	ND	ND
JAN. 10	ND	ND
APR. 09	ND	ND
MAR. 08	ND	ND
JUN. 07	ND	0.2 J
AUG. 06	NS	NS
JAN. 06	NS	NS
APR. 96	ND	ND
NOV. 95	ND	ND

MW25-19

DATE	BTEX	TOT. CHLOR. ORGS.
MAR. 16	ND	ND
MAR. 15	ND	ND
JUN. 14	5.5	ND
MAY 13	0.82 J	ND
FEB. 12	0.68J	ND
FEB. 11	ND	ND
AUG. 10	ND	ND
JAN. 10	ND	ND
APR. 09	1.7	ND
MAR. 08	ND	ND
JUN. 07	NS	NS
AUG. 06	ND	ND
JAN. 06	ND	ND
APR. 96	ND	ND
NOV. 95	15 J	ND
JAN. 93	138 J	5 J

MW25-8

DATE	BTEX	TOT. CHLOR. ORGS.
MAR. 16	NA	NA
MAR. 15	NA	NA
JUN. 14	NA	NA
MAY 13	ND	ND
FEB. 12	ND	ND
FEB. 11	NS	NS
AUG. 10	NS	NS
JAN. 10	ND	ND
APR. 09	ND	ND
MAR. 08	ND	ND
JUN. 07	NS	NS
AUG. 06	ND	ND
JAN. 06	ND	ND
APR. 96	ND	ND
NOV. 95	ND	ND

MW25-15

DATE	BTEX	TOT. CHLOR. ORGS.
MAR. 16	NA	NA
MAR. 15	NA	NA
JUN. 14	NA	NA
MAY 13	ND	ND
FEB. 12	ND	ND
FEB. 11	ND	ND
AUG. 10	ND	ND
JAN. 10	ND	ND
APR. 09	ND	ND
MAR. 08	ND	ND
JUN. 07	NS	NS
AUG. 06	ND	ND
JAN. 06	ND	ND
APR. 96	0.6	ND
NOV. 95	ND	ND

MW25-17

DATE	BTEX	TOT. CHLOR. ORGS.
MAR. 16	ND	ND
MAR. 15	ND	ND
JUN. 14	ND	ND
MAY 13	ND	ND
FEB. 12	ND	ND
FEB. 11	ND	ND
AUG. 10	ND	ND
JAN. 10	ND	ND
APR. 09	ND	ND
MAR. 08	ND	ND
JUN. 07	ND	ND
AUG. 06	ND	ND
JAN. 06	ND	ND
MAR. 96	ND	ND
NOV. 95	ND	ND

AREA OF REMOVAL ACTION
FIRE TRAINING AND DEMONSTRATION PAD

SB25-4
SB25-1
SB25-5

MW25-3
MW25-2

CRUSHED SHALE ROAD

DATE	BTEX	TOT. CHLOR. ORGS.
MAR. 16	NA	NA
MAR. 15	NA	NA
JUN. 14	NA	NA
MAY 13	ND	ND
FEB. 12	ND	ND
FEB. 11	ND	ND
AUG. 10	ND	ND
JAN. 10	ND	ND
APR. 09	ND	ND
MAR. 08	ND	ND
JUN. 07	ND	ND
AUG. 06	ND	ND
JAN. 06	ND	ND
MAR. 96	ND	ND
NOV. 95	ND	ND

MW25-18

MW25-9

MW25-10

SEAD25

CONDUIT

DATE	BTEX	TOT. CHLOR. ORGS.
MAR. 16	1.05	ND
MAR. 15	0.64 J	ND
JUN. 14	42.2	7.48
MAY 13	31.9	6.52
MAR. 12	1.46 J	1.21 J
FEB. 11	37.5 J	16.9 J
AUG. 10	115.6 J	24.8 J
JAN. 10	4 J	2.4 J
APR. 09	30.8	4.9 J
MAR. 08	1.89 J	ND
JUN. 07	28.9 J	2.01 J
AUG. 06	2.77 J	ND
APR. 06	35	ND
APR. 96	6220	89 J
NOV. 95	3040	68 J
FEB. 94	3950	96 J

ADMINISTRATION AVENUE

DATE	BTEX	TOT. CHLOR. ORGS.
MAR. 16	ND	ND
MAR. 15	ND	1.6
JUN. 14	NS	NS
MAY 13	0.34 J	ND
FEB. 12	0.4J	ND
FEB. 11	0.74 J	ND
AUG. 10	NS	NS
JAN. 10	ND	ND
APR. 09	0.46 J	ND
MAR. 08	4.62 J	ND
JUN. 07	NS	NS
AUG. 06	0.58 J	ND
JAN. 06	124	5.44
APR. 96	40 J	2 J
NOV. 95	165	10 J

ORDNANCE

MW25-13

DATE	BTEX	TOT. CHLOR. ORGS.
MAR. 16	NA	NA
MAR. 15	NA	NA
JUN. 14	NA	NA
MAY 13	NS	NS
FEB. 12	ND	ND
FEB. 11	NS	NS
AUG. 10	NS	NS
JAN. 10	NS	NS
APR. 09	NS	NS
MAR. 08	ND	ND
JUN. 07	NS	NS
AUG. 06	ND	ND
JAN. 06	ND	ND
MAR. 96	ND	ND
NOV. 95	ND	ND

DATE	BTEX	TOT. CHLOR. ORGS.
MAR. 16	ND	ND
MAR. 15	ND	ND
JUN. 14	NS	NS
MAY 13	ND	ND
FEB. 12	ND	ND
FEB. 11	ND	ND
AUG. 10	NS	NS
JAN. 10	ND	ND
APR. 09	NS	NS
MAR. 08	ND	ND
JUN. 07	NS	NS
AUG. 06	ND	0.53 J
JAN. 06	ND	ND
MAR. 96	ND	ND
NOV. 95	ND	ND

LEGEND

- DRAINAGE DITCH
- FENCE
- UNPAVED ROAD
- SEAD 25 BOUNDARY
- BRUSH LINE
- RAILROAD
- 760 — GROUND SURFACE ELEVATION CONTOUR
- UNDERGROUND ELECTRIC UTILITY LINE
- UNDERGROUND WATER UTILITY LINE
- ROAD SIGN
- OVERHEAD UTILITY POLE
- HYDRANT
- MANHOLE
- UTILITY BOX
- DECIDUOUS TREE
- COORD. GRID (250' GRID)
- POLE
- SEAD-25 — SURVEY MONUMENT
- MW25-2 — MONITORING WELL DESIGNATION
- NOV/DEC 2005 REMEDIATED AREAS
- SB25-3 — PRE-EXCAVATION SOIL SAMPLING BORING LOCATIONS

DATE	BTEX	TOT. CHLOR. ORGS.
JUN. 07	29.9	2.01
AUG. 06	3	ND
APR. 06	35	ND
NOV. 95	3040	68
FEB. 94	3950	71

CONTAMINANT CONCENTRATIONS OF BTEX:
- BENZENE
- TOLUENE
- ETHYL BENZENE
- TOTAL XYLENES (OR M/P + O XYLENE)

AND TOTAL CHLORINATED ORGANICS:
- 1,1,1-TRICHLOROETHANE
- 1,1-DICHLOROETHANE
- 1,2-DICHLOROETHENE TOTAL (OR 1,2-DICHLOROETHENE)
- CIS-1,2-DICHLOROETHENE
- CHLOROFORM
- TRICHLOROETHENE
- VINYL CHLORIDE

UNITS (ug/L)
APR 96, NOV 95, FEB 94, & JAN 93 ARE PRE-REMEDIATION.
ALL OTHER ROUNDS (BOLD) ARE POST-REMEDIATION.

- ND — NON-DETECT
- NS — NOT SAMPLED DUE TO LOW GROUNDWATER LEVELS
- NA — WELL NO LONGER SAMPLED AS PART OF THE LTM PROGRAM

NOTES:

1. THE TOTAL BTEX OR TOTAL CHLORINATED ORGANICS CONCENTRATION IS THE SUM OF DETECTED VALUES ONLY.
2. AT WELL LOCATIONS WHERE A DUPLICATE SAMPLE WAS COLLECTED, THE AVERAGE RESULT OF THE SAMPLE AND THE DUPLICATE IS PRESENTED.
3. BEGINNING WITH THE JUNE 2014 SAMPLING EVENT, THE NUMBER OF WELLS SAMPLED AS PART OF THE LTM WAS REDUCED FROM 10 WELLS TO 5 WELLS CONSISTING OF MW25-2, MW25-3, MW25-9, MW25-10, AND MW25-17.

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT

ROMULUS, NEW YORK

2016 ANNUAL LONG-TERM MONITORING REPORT FOR SEAD-25

DEPT. ENVIRONMENTAL ENGINEERING | Dwg. No.

FIGURE 6

VOCS DETECTED IN GROUNDWATER AT SEAD-25

SCALE AS SHOWN | DATE JUNE 2016 | REV

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\SEAD 25\ANNUAL RPT 9 2016\DRAFT\FIGURES\FIGURE FILES-ACAD ETC\FIGURE 6.DWG, DATE: 05/26/2016 10:25:46AM, p0018397

Figure 7A
Concentrations of BTEX over Time at MW25-2
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

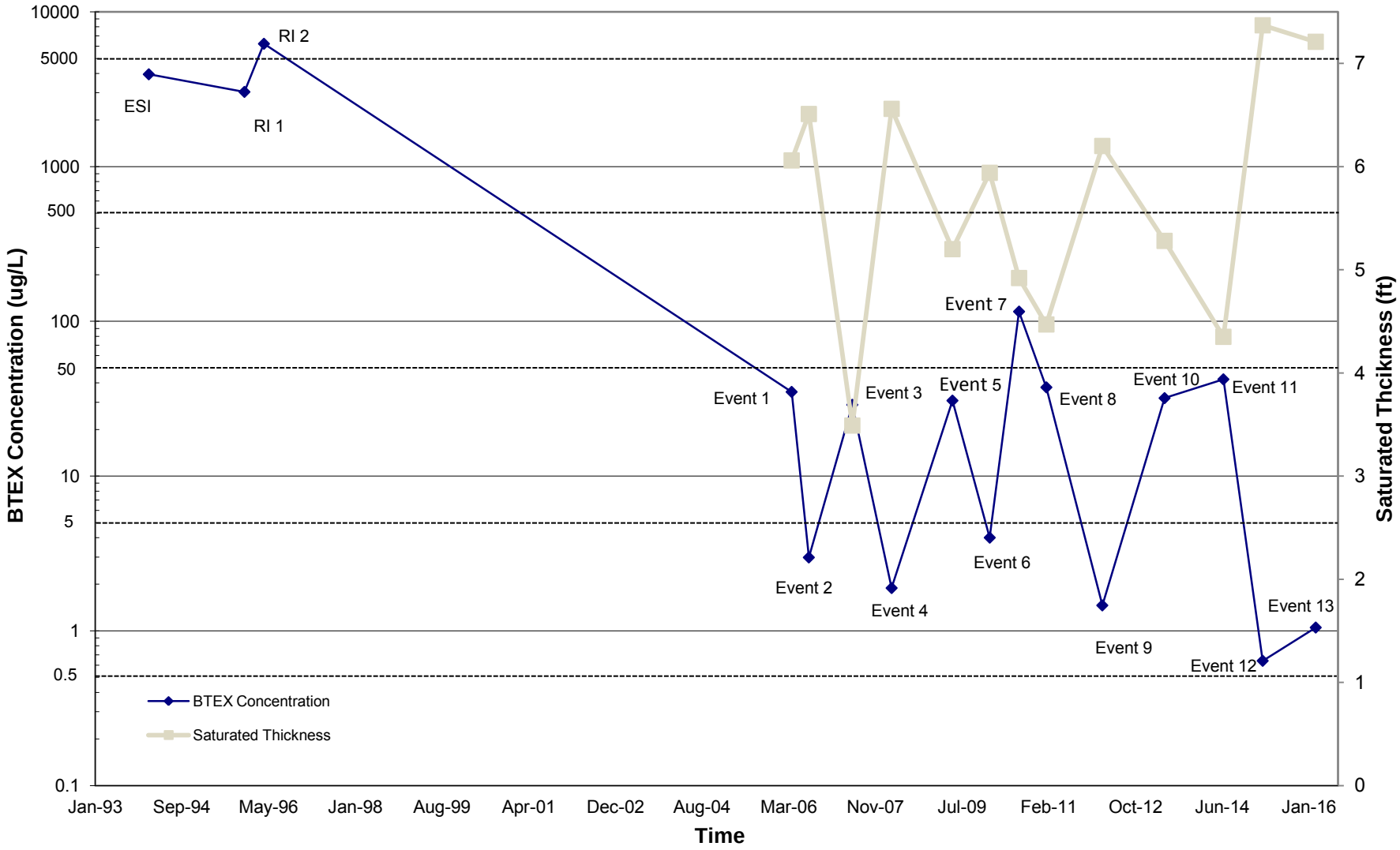


Figure 7B
Concentrations of BTEX over Time at MW25-3
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

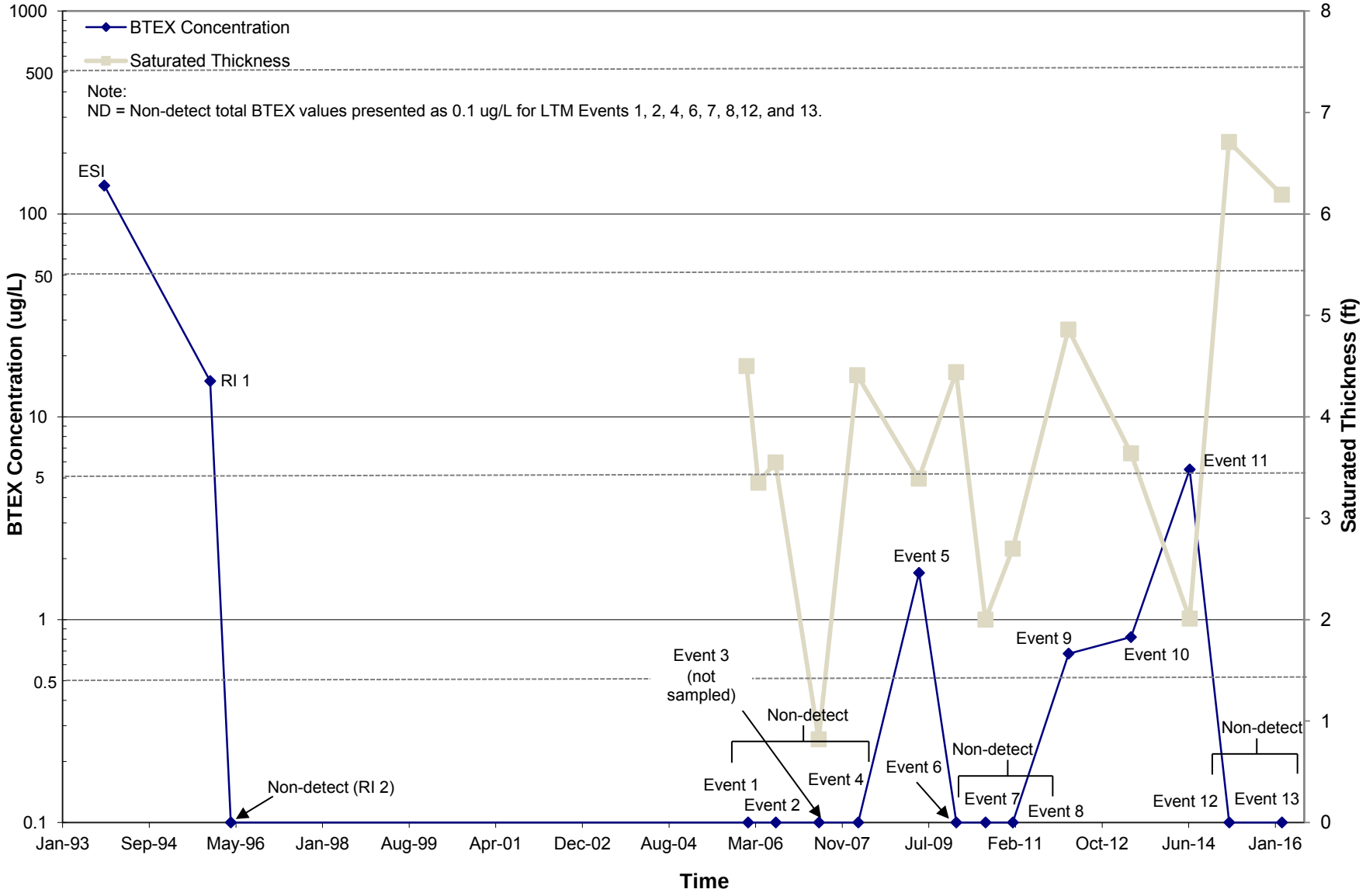


Figure 7C
Concentrations of BTEX over Time at MW25-9
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

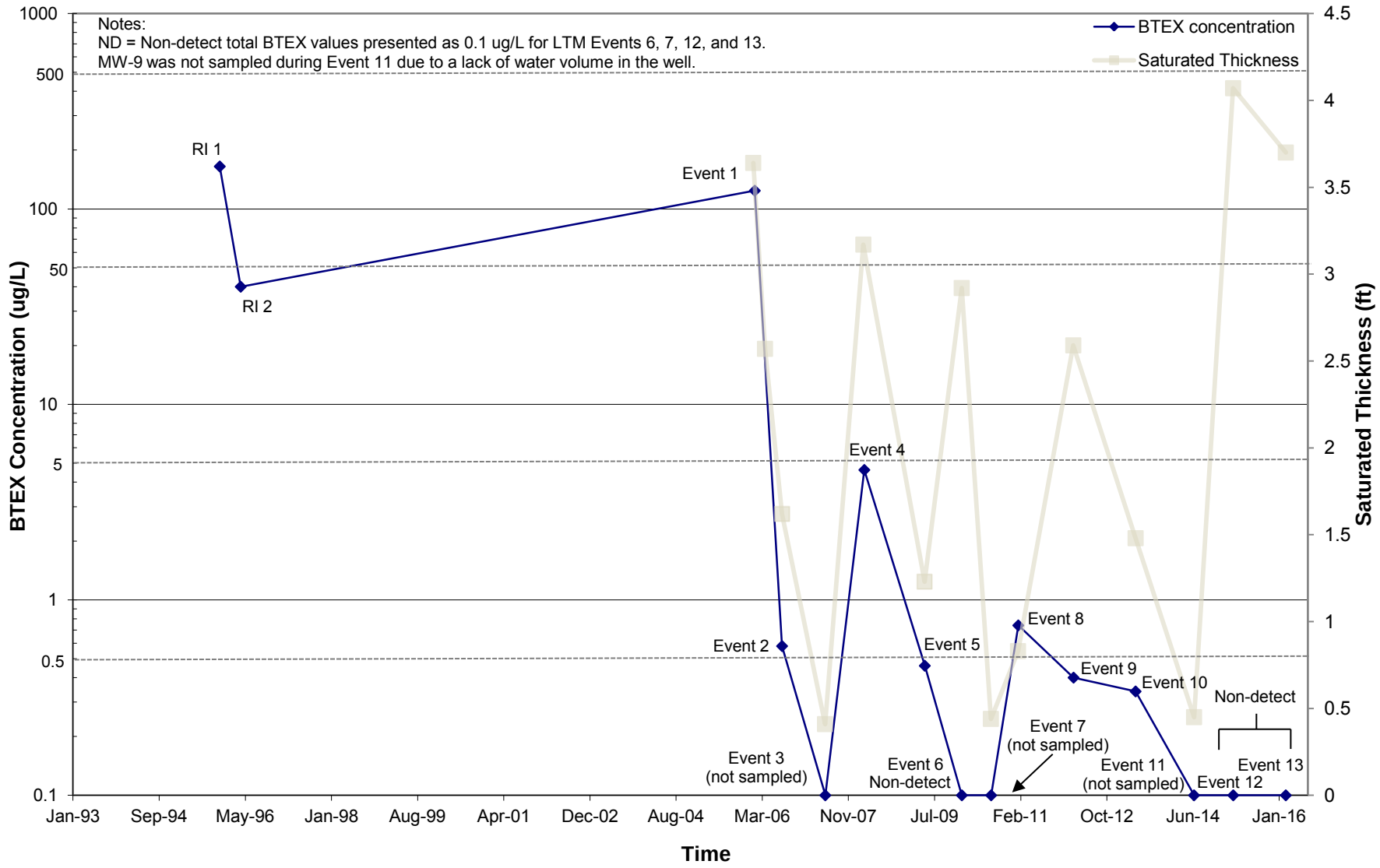


Figure 8A
Chlorinated VOC COC Concentrations at MW25-2
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

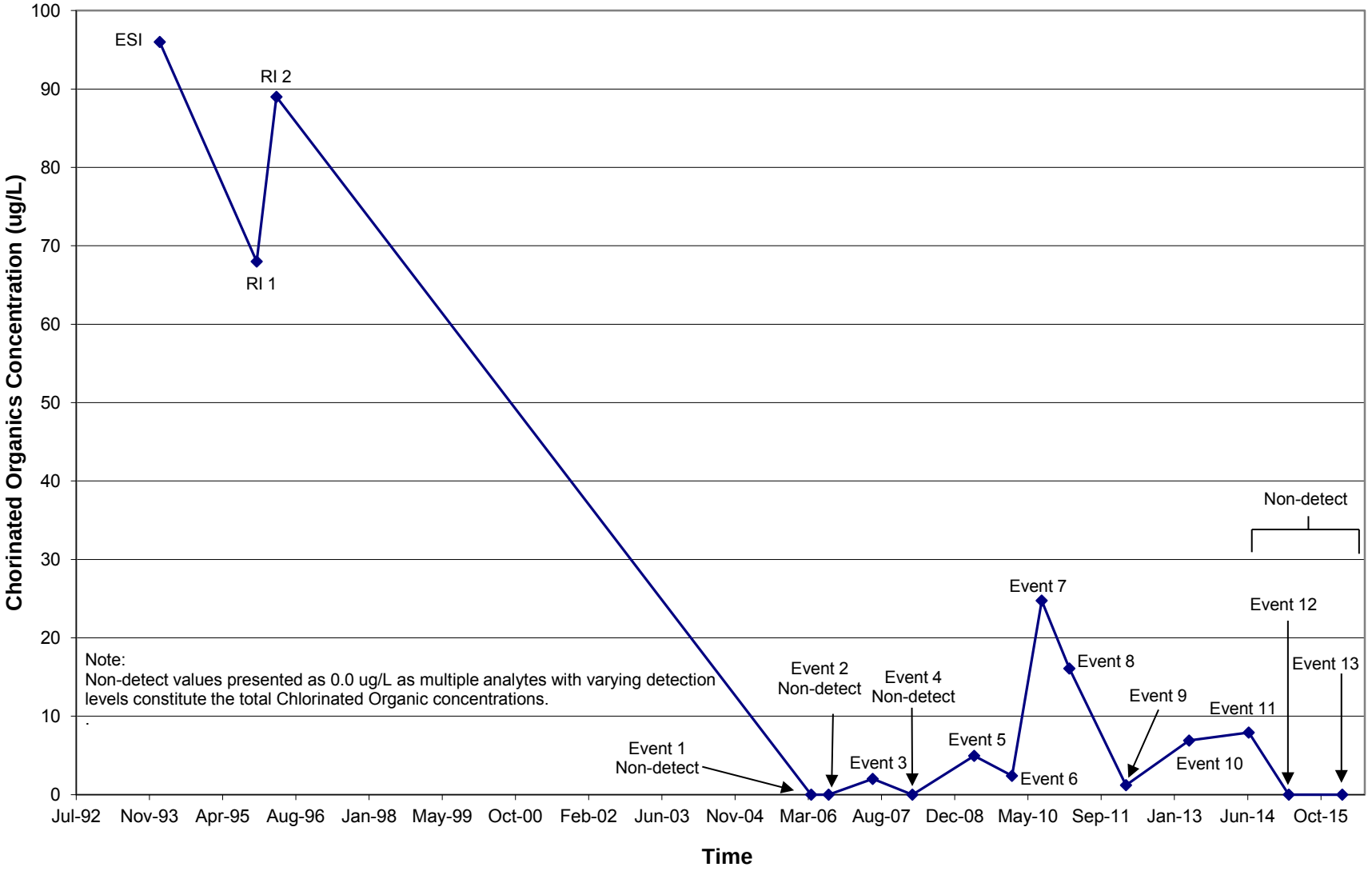


Figure 8B
Chlorinated VOC COC Concentrations at MW25-3
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

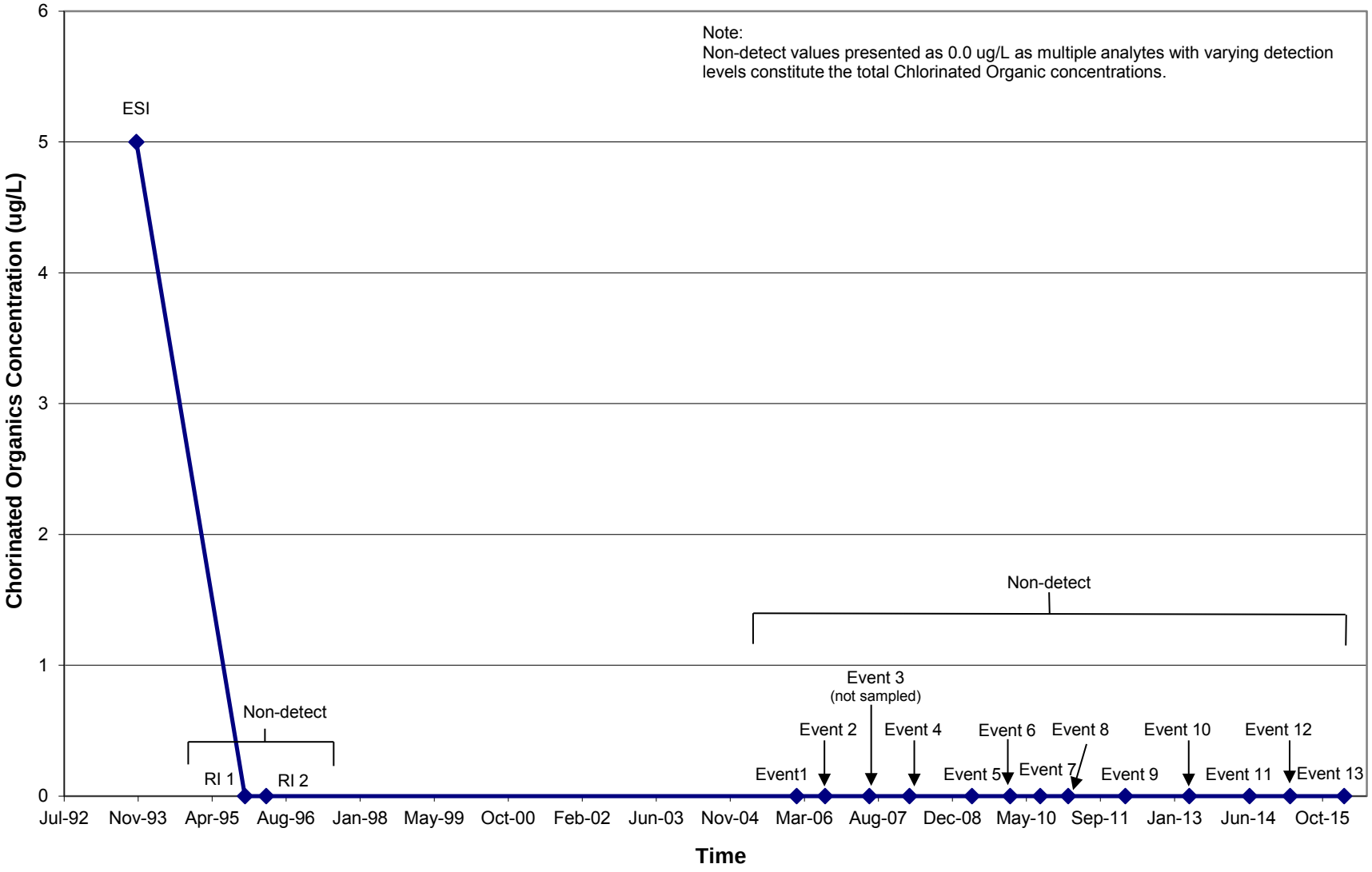


Figure 8C
Chlorinated VOC COC Concentrations at MW25-9
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

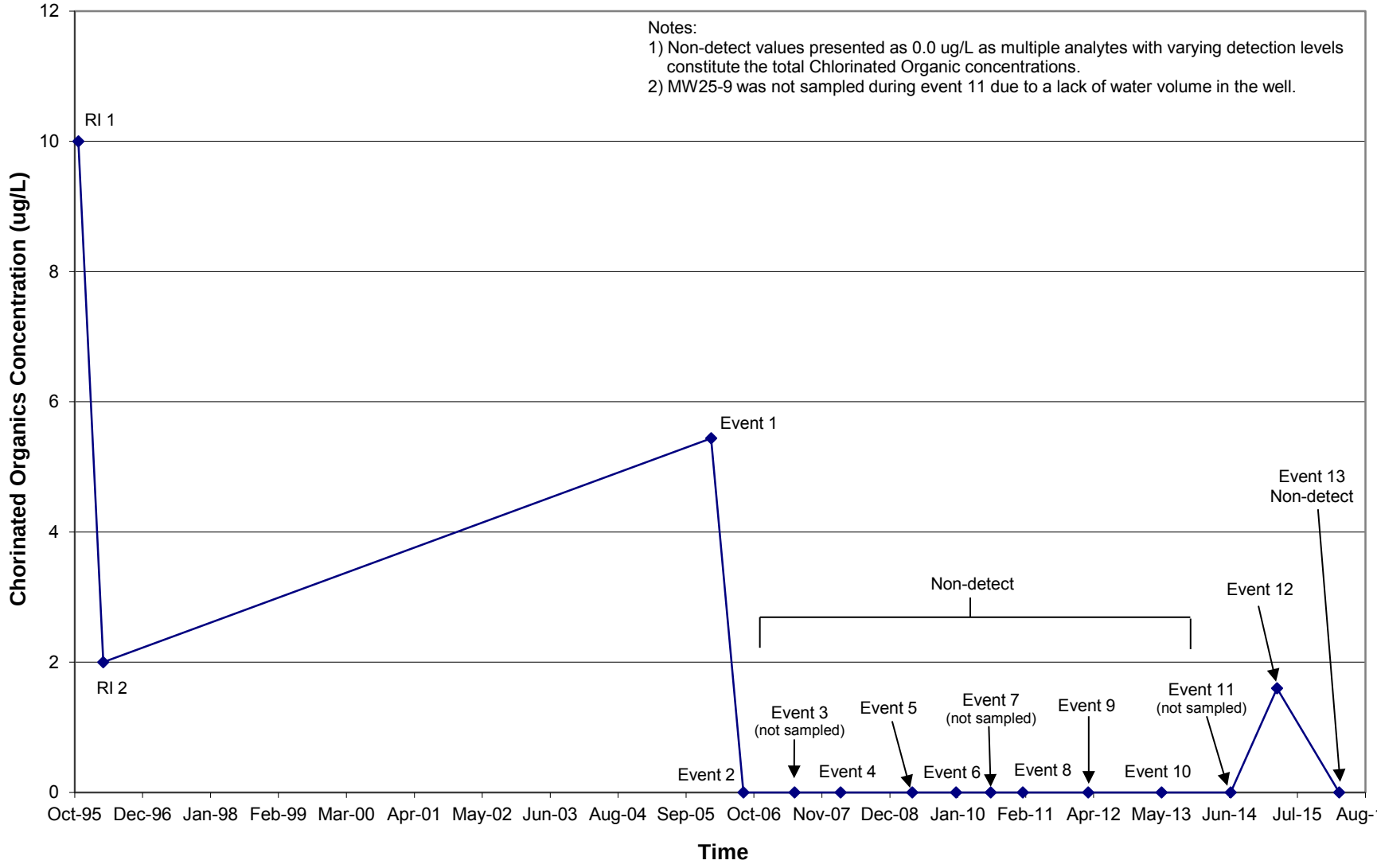


Figure 9A
Concentrations of Detected COCs in MW25-2
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activiy

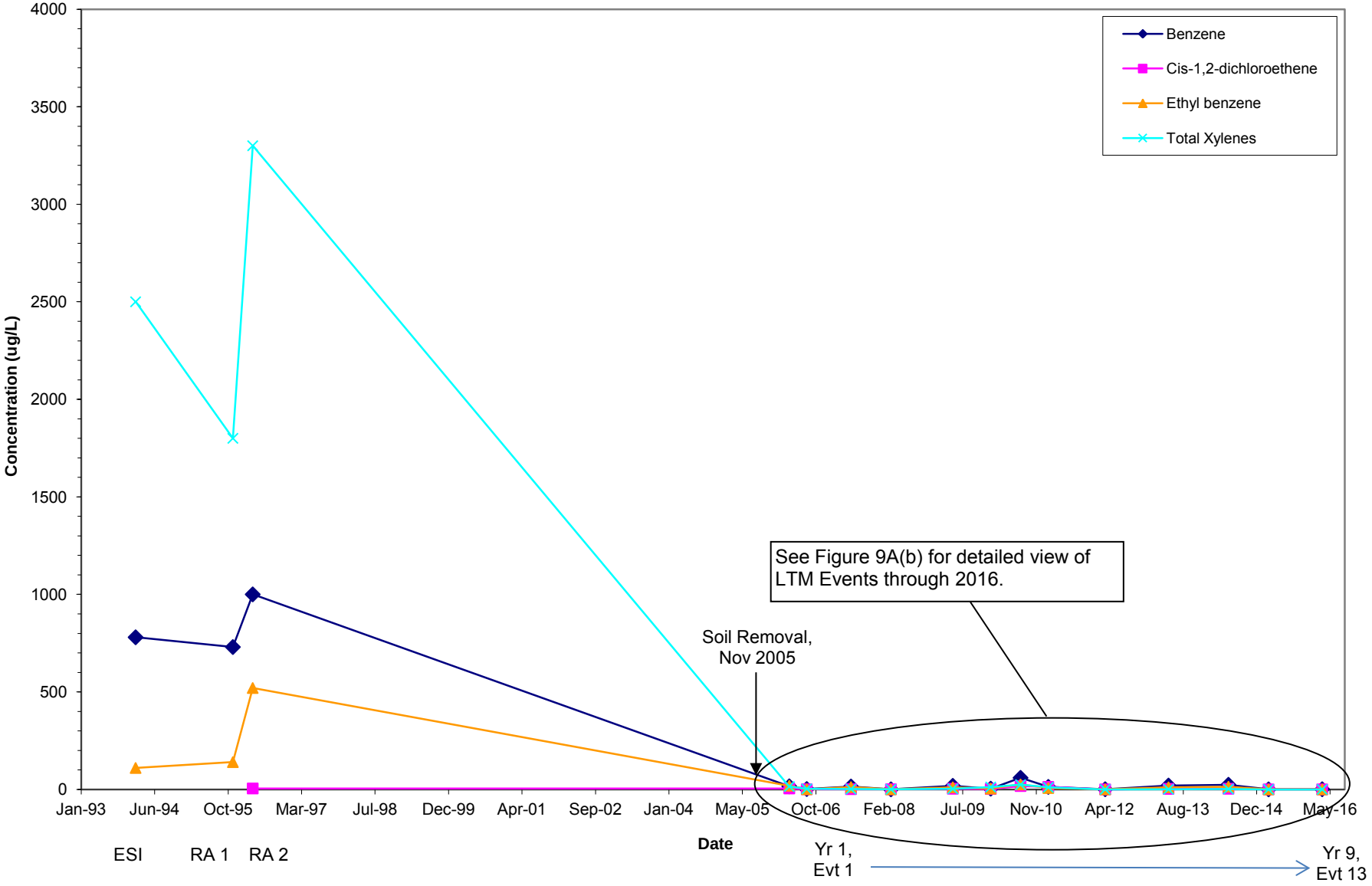


Figure 9A(b)
Concentrations of Detected COCs in MW25-2
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activiy

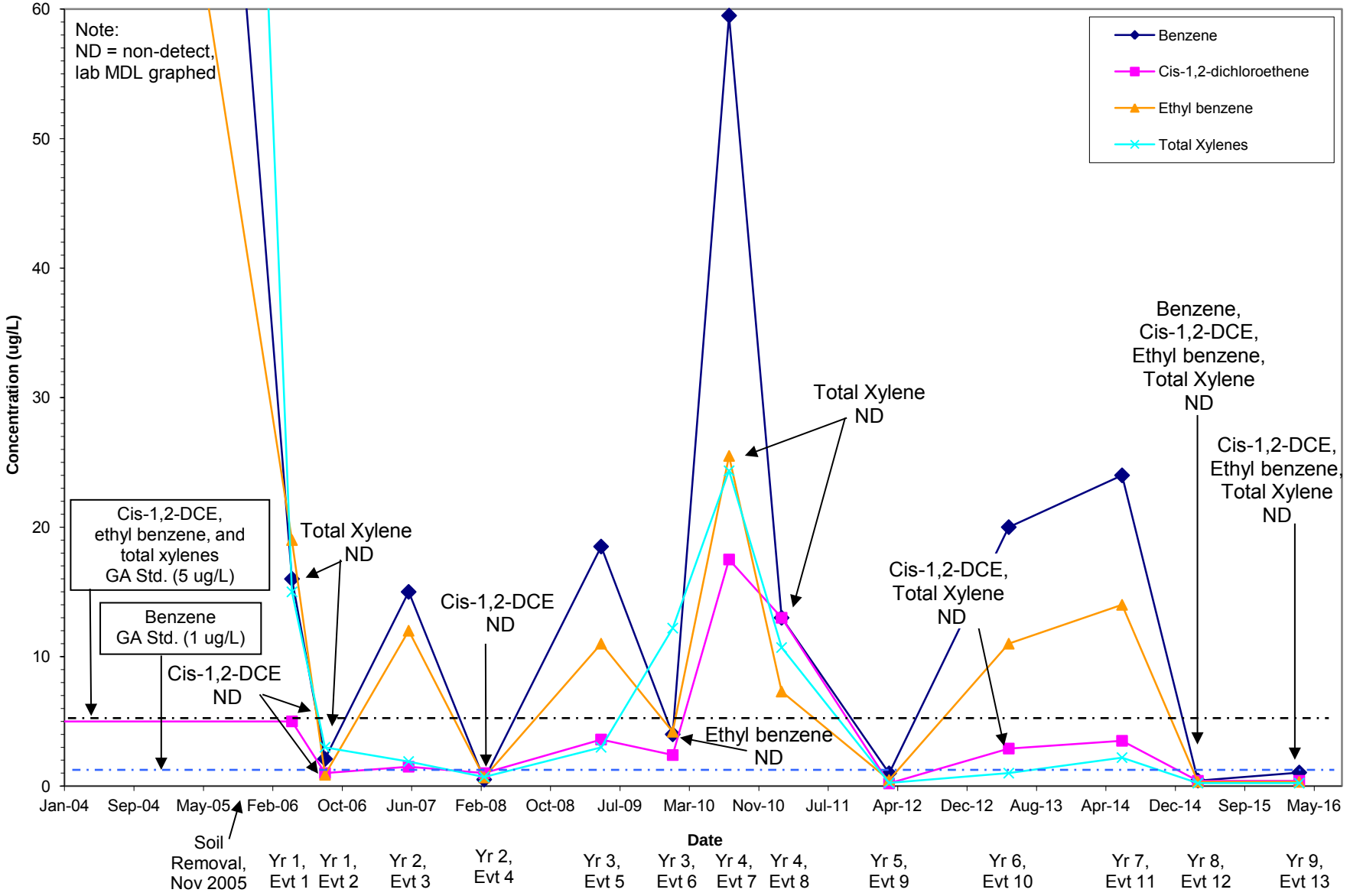


Figure 9B
Concentrations of Detected COCs in MW25-3
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activiy

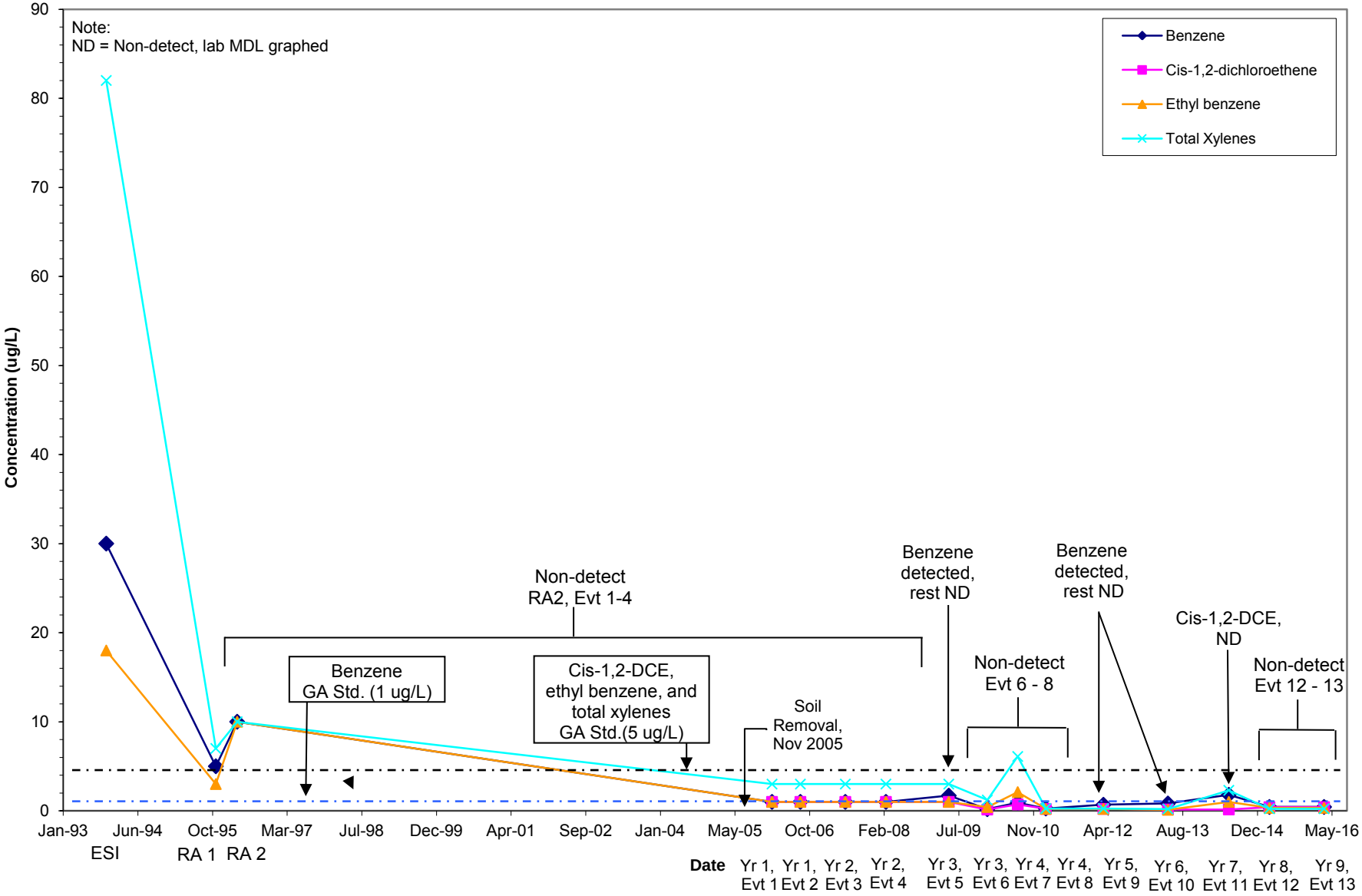
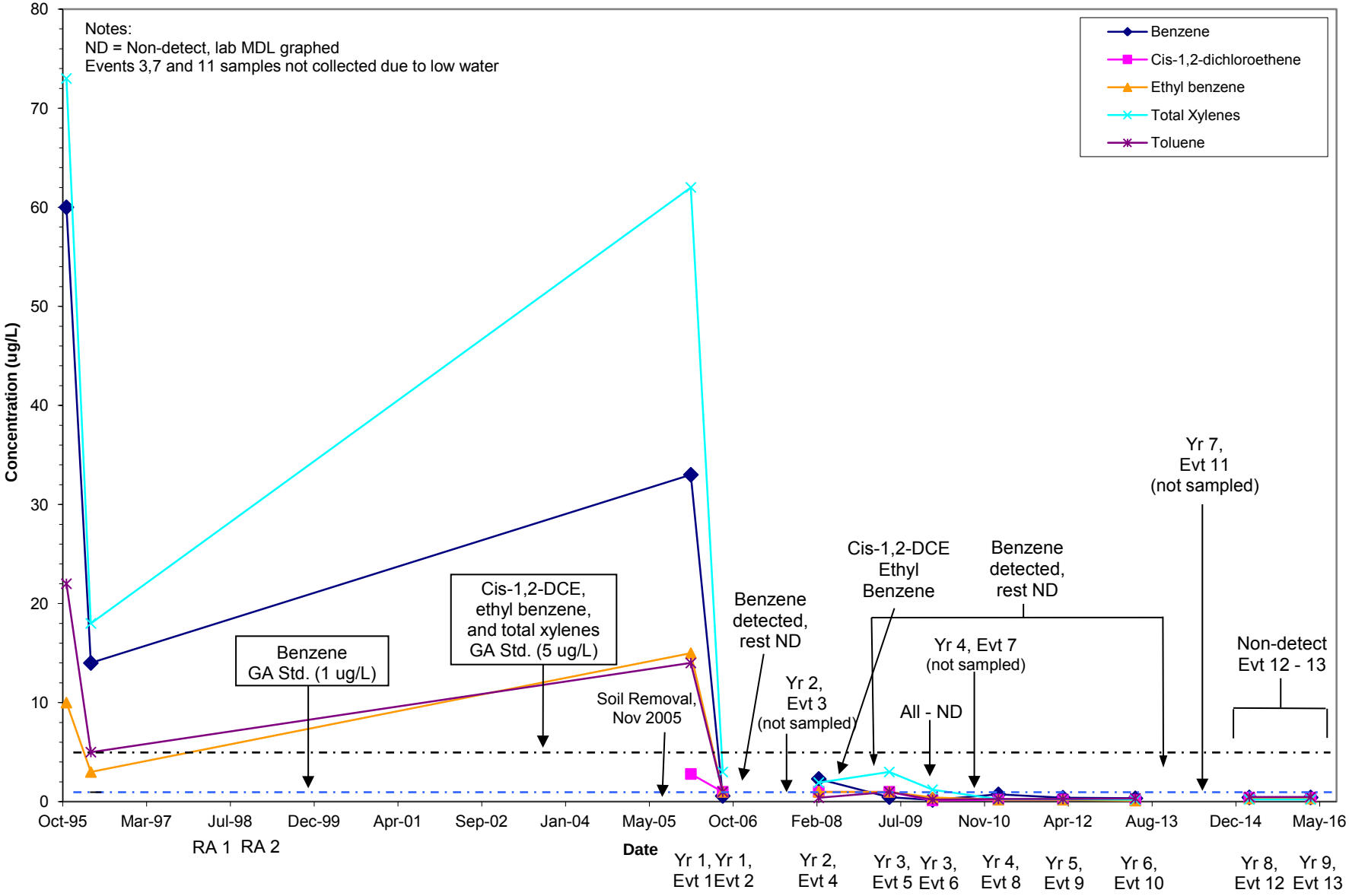


Figure 9C
Concentrations of Detected COCs in MW25-9
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activiy



APPENDICES

A Long-Term Monitoring Event 2016 Field Forms

B Long-Term Monitoring Event 2016 Laboratory Reports

C Historic Groundwater Elevations (Events 1 through 13)

D Complete LTM Groundwater Analytical Data (Events 1 through 13)

E Long-Term Monitoring Event 2016 Data Validation Sheets

APPENDIX A

LONG-TERM MONITORING EVENT 2016 FIELD FORMS

PAGE 1 OF 1

GROUNDWATER ELEVATION REPORT

PARSONS | **CLIENT:** | **DATE:** 3/16/16

PROJECT: SEAD-25 LTM Rnd 13
LOCATION: ST
PROJECT NO:
INSPECTOR: 320

MONITORING EQUIPMENT:

INSTRUMENT	DETECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR:

INSTRUMENT	CORRECTION FACTOR

COMMENTS: 0.3 ft probe sensor tip length

WELL	TIME	DEPTH TO WATER	DEPTH TO ~~PRODUCT~~ Well	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC. GRAV.	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of: riser, concrete, protective casing, etc.)
25-1	1245	5.48	7.55					wasp nest in well, locked, well hinge cracke
25-13	1257	2.43	5.25					No well cap, locked, wasp nests
25-15	~~1300~~ 1301	2.83	6.95					locked, ants, brush around well
25-19	1307	3.2	11.8					locked, well cap
25-6	1312	3.20	13.7					locked, well cap
~~25-18~~ 25-18	1316	4.0	11.0					locked, well cap
25-17	1319	2.10	10.72					locked, well cap, brush around well
25-9	1323	1.8	5.2					locked, well cap
25-8	1325	1.8	5.2					not locked, PVC lifted, well cap
25-10	1328	2.52	6.15					locked, well cap
25-3	1331	3.66	9.55					locked, PVC lifted, barely opens, well cap
25-2	1337	~~7.14~~ 4.14	11.05					locked, well cap
								locked, well cap

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW25-2

PROJECT: SEAD-25 LTM Groundwater Sampling - Round 13
LOCATION: ROMULUS, NY

DATE: 3/17/16
INSPECTORS: BBO/DD
PUMP #: 12867
SAMPLE ID #: 25LM20119 / 25LM20120

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND (FROM) VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS
1354	50s	Scattered cloud		10-15	W→E	

MONITORING INSTRUMENT	MONITORING DETECTOR
OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
1.12 gal = 1 well vol / 3.36 gal 3 well vol

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	11.05					
DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)		DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		4.18'				
RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)			PUMP AFTER SAMPLING (cps)		

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (cm/S^2)	pH	ORP (mV)	TURBIDITY (NTU)
1355	4.08	Bladder Pump & YSI in cell							
1356		Pump Started		YSI	YSI	Horiba	→	Horiba	Hach
1402	4.80	~134		0.25	5.4	0.713	7.84	49	16.4
1407	4.85			0.27	5.3	0.726	7.79	27	12.8
1412	4.98	~130		0.32	5.1	0.731	7.76	12	11.5
1417	4.95		~0.5 gal	0.30	5.1	0.720	7.76	3	7.59
1422	4.99			0.28	5.1	0.724	7.75	-1	4.65
1427				0.33	5.1	0.719	7.75	-5	3.13
1432	5.02		~1.0 gal	0.54	5.1	0.715	7.75	-9	2.77
1437	5.03			0.69	5.1	0.705	7.74	-13	3.11
1442	5.03		~1.5 gals	0.90	5.2	0.696	7.73	-15	3.40
1447	5.05			0.81	5.1	0.687	7.74	-17	4.40
1452	5.08		~1.75 gals	0.61	5.1	0.669	7.74	-17	3.67
1500		Samples Collected		SA, DU, MS, MSD		filled bottle sets of:			
						3x VOAs for	VOC		
		Sulfide 0.03		mg/L		3x VOAs for	MEE		
						1x 125ml	NO2/NO3 Preserved		
			~2.25 gals			1x 250ml	NO2/NO3 Unpreserved		
						1x 125ml	Cl/Sulfide		
						1x 250ml	Fe/Na		

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW25-3

PROJECT: SEAD-25 LTM Groundwater Sampling - Round 13
LOCATION: ROMULUS, NY

DATE: 3/17/16
INSPECTORS: BDO / DD
PUMP #: 30971
SAMPLE ID #: 25LM20121

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS
1219	50's	Partly Cloudy		5-15	W→E	concrete

MONITORING INSTRUMENT	MONITORING DETECTOR
OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
0.4 = 1 well Vol / 1.22 = 3 well Vol

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	6.23'					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		3.72			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (cm/S^2)	pH	ORP (mV)	TURBIDITY (NTU)
1224	3.35	Pump & YSI in well							
1225		Pump Started	YSI	YSI	Horiba	→	Horiba		Hach
1227		Pulled pump due to bubbles Sealed well, cut lines & reduced # O-Rings							
1231		Pump re-started							
1238	4.06			1.66	5.2	0.666	7.98	169	33.5
1242	4.24	~132		1.84	5.0	0.706	7.90	174	61.3
1247	4.53	~144		1.77	4.8	0.713	7.87	175	33.4
1253	4.63	~130		1.90	4.7	0.727	7.86	162	15.5
1258	4.67	~120		2.41	4.6	0.724	7.84	116	9.24
1303	4.67			2.15	4.6	0.717	7.83	65	6.67
1308	4.69			1.88	4.5	0.715	7.82	35	4.45
1312	4.73	124	~1.0 gal	1.42	4.4	0.709	7.82	18	3.77
1317	—			1.14	4.4	0.700	7.81	4	3.57
1322	4.72			1.08	4.4	0.700	7.81	-4	2.81
1327	4.76		1.5 gals	0.95	4.4	0.689	7.81	-15	2.49
1335		Sample Collected				3x VOAs for VOC			
						3x VOAs for MEE			
		Sulfide = 0.10	~3/L			1x 125ml NO2/NO3 Preserved			
						1x 250ml NO2/NO3 Unpreserved			
						1x 125ml Cl/Sulfide			
						1x 250ml Fe/Na			

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW25-9
PROJECT: SEAD-25 LTM Groundwater Sampling - Round 13		DATE: 3/17/16
LOCATION: ROMULUS, NY		INSPECTORS: BBO / DD
		PUMP #: 20951
		SAMPLE ID #: 25LM20122

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS
857	50's	Sunny Scattered clouds		5-10	SW→NE	wet overnight rain

MONITORING INSTRUMENT	MONITORING DETECTOR
OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

0.57 gal = 1 well / 1.73 gal 3 well

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	5.2					
DATA COLLECTED AT WELL SITE	**PID READING (OPENING WELL)**	**DEPTH TO STATIC WATER LEVEL (TOC)**		**DEPTH TO STABILIZED WATER LEVEL (TOC)**	**DEPTH TO PUMP INTAKE (TOC)**	**PUMPING START TIME**
		1.66'				
RADIATION SCREENING DATA	**PUMP PRIOR TO SAMPLING (cps)**			**PUMP AFTER SAMPLING (cps)**		

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (cm/S^2)	pH	ORP (mV)	TURBIDITY (NTU)
912	1.20	Bladder Pumps & YSI in well							
913		Pump	Started	YSI	YSI	Horiba →	Horiba	Hach	Hach (not connected right)
~~918~~	~~1.85~~			~~2.52~~	~~5.6~~	~~0.00~~	~~7.31~~	~~191~~	flowcell
927	2.68	~130		3.08	5.6	0.512	8.55	215	
932	2.83	~110		2.50	5.5	0.513	8.43	224	4.01
937	2.80	~110	~0.5 gal	1.00	5.3	0.511	8.33	224	2.18
942	2.83			0.59	5.2	0.508	8.27	222	1.65
947	2.81			0.62	5.3	0.502	8.24	220	1.95
952	2.82	~110	~0.75 gal	0.63	5.2	0.499	8.15	218	1.97
957	2.73			1.18	5.1	0.497	8.18	215	1.30
1002	2.74		~1.0 gal	1.09	5.0	0.492	8.16	212	1.68
1007	2.85	~100		0.74	5.1	0.491	8.14	209	1.35
1012	2.85			0.91	5.0	0.486	8.12	206	1.22
1017	2.80		~1.25 gal	0.70	5.0	0.483	8.10	203	—
1022	2.69			0.62	5.0	0.470	8.06	202	2.27
1028	2.62		~1.5 gal	0.83	4.9	0.470	8.06	199	2.07
1035		Samples	Collected			3x VOAs for VOC			
			~1.6 gal			3x VOAs for MEE			
			Sulfide =	0.04 mg/L		1x 125 ml NO2/NO3 Preserved			
						1x 250 ml NO2/NO3 Un Preserved			
						1x 125 ml Cl/Sulfate			
						1x 250 ml Fe/Na			

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW25-10

PROJECT: SEAD-25 LTM Groundwater Sampling - Round 13
LOCATION: ROMULUS, NY

DATE: 3/17/16
INSPECTORS: BDC / DD
PUMP #: 9500
SAMPLE ID #: 25LM20123

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS
1059	55	Sunny		5-10	SW-NE	overcast rain

MONITORING INSTRUMENT	DETECTOR
OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
HISTORIC DATA	6.16					
	PID READING (OPENING WELL)	**DEPTH TO STATIC WATER LEVEL (TOC)**		**DEPTH TO STABILIZED WATER LEVEL (TOC)**	**DEPTH TO PUMP INTAKE (TOC)**	**PUMPING START TIME**
DATA COLLECTED AT WELL SITE		2.63				
RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)			PUMP AFTER SAMPLING (cps)		

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (cm/S^2)	pH	ORP (mV)	TURBIDITY (NTU)
1100	~~2.73~~	YSI & Bladder Pump on well							
	2.57								
1103		Pump Started		YSI	YSI	Horiba	→	Horiba	Hach
1113	3.2	90		7.82	6.0	0.341	8.12	168	6.40
1118	3.37	90		~~7.87~~	5.9	0.337	8.08	174	4.73
1123	3.46			8.07	5.9	0.345	8.07	175	2.92
1128	3.54	106		7.96	5.9	0.363	8.07	175	2.45
1133	3.75			8.24	6.0	0.377	8.06	175	2.89
1137	3.78	106	~0.6 gal	8.42	5.9	0.380	8.05	175	1.69
1143	3.72			8.19	5.8	0.380	8.02	176	1.46
1147	3.75	96	~1.0 gal	8.23	5.8	0.386	8.01	175	1.21
1153	3.78			8.36	5.7	0.389	7.99	175	1.13
1200		Sample Collected				3x VOAs for VOC			
						3x VOAs for MEE			
		Sulfide 0.01 mg/L				1x 125 ml NO2/NO3 Preserved			
						1x 250 ml NO2/NO3 Unpreserved			
						1x 125 ml Cl/Sulfide			
						1x 250 ml Fe/Na			

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW25-17

PROJECT: SEAD-25 LTM Groundwater Sampling - Round 13
LOCATION: ROMULUS, NY

DATE: 3/16/16
INSPECTORS: BBO
PUMP #: 16362
SAMPLE ID #: 25LM20118

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS
1343		rainy shower				

MONITORING

INSTRUMENT	DETECTOR
OVM-580	PID

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
~1.4 gal 1 well vol / 4.2 gal 3 well vol

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
11.07	10.72'					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		2.10			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (cm/S^2)	pH	ORP (mV)	TURBIDITY (NTU)
1407	1.75	YSI?	Pump in well						
1408		Pump	Started	YSI	YSI	Horiba		Horiba	Hach
1435	2.92		~125	13.47	5.7	0.491	8.72	146	4.88
1440	3.03	~144		12.69	5.6	0.487	8.61	157	2.02
1445	2.82			12.25	5.6	0.481	8.54	159	2.74
1450		~50		11.98	5.6	0.486	8.48	160	2.07
1455	2.66	~80	~0.5	11.64	5.6	0.487	8.45	161	1.61
1500	2.75	~102		11.13	5.6	0.472	8.43	163	1.17
1505	2.87	~100		10.75	5.6	0.486	8.41	164	0.95
1510	2.88	~98	~1.0 gal	10.53	5.6	0.485	8.40	165	0.90
1515	2.85			8.53	5.6	0.483	8.40	166	1.49
1520		~110	~1.25 gal	6.86	5.5	0.485	8.39	167	2.48
1525	2.95			6.53	5.6	0.485	8.38	168	0.97
1530	2.90		~1.60	6.53	5.7	0.482	8.38	169	1.44
1535	2.94	~112		6.37	5.7	0.481	8.37	170	0.92
1540	2.97		~1.75 gal	6.24	5.6	0.482	8.37	170	0.75
1547		Samples Collected				3x Vol for VOC			
			~2.1 gals			3x Vols for MEE			
		Sulfide 0.01 mg/L				1x 125 ml NO2/NO3 Preserved			
						1x 250 ml NO2/NO3 Unpreserved			

1X 125 ml Cl/Sulfate
1X 250 ml Fe/Na

APPENDIX B

LONG-TERM MONITORING EVENT 2016 LABORATORY REPORTS

Laboratory Reports are provided on the electronic (CD) version of this report.

APPENDIX C

HISTORIC GROUNDWATER ELEVATIONS (EVENTS 1 THROUGH 13)

Appendix C
Historic Groundwater Elevations (Events 1 through 13)
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

					Event 1 - January 2006				Event 1 - April 2006				Event 2 - August 2006			
Monitoring Well	Top of Risor Elevation (ft)	Well Depth (ft)	4/29/09 Revised Top of Risor Elevation (ft) [3]	Well Depth (Post-2008) (ft)	Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)
MW25-1	743.00	7.77	743.00	7.77	1/20/06	2.10	5.67	737.33	4/12/06	1.97	5.80	737.20	8/9/06	2.12	5.65	737.35
MW25-2	746.36	11.31	746.36	11.31	1/20/06		NA		4/12/06	6.06	5.25	741.11	8/9/06	6.51	4.8	741.56
MW25-3	745.76	9.00	746.34	9.58	1/20/06	4.50	4.50	741.26	4/12/06	3.35	5.65	740.11	8/9/06	3.55	5.45	740.31
MW25-6	744.44	14.27	744.44	14.27	1/20/06	10.02	4.25	740.19	4/12/06	8.77	5.50	738.94	8/9/06	8.57	5.7	738.74
MW25-8	742.46	5.47	742.46	5.47	1/20/06	3.67	1.80	740.66	4/12/06	2.67	2.80	739.66	8/9/06	2.27	3.2	739.26
MW25-9	742.36	5.42	742.36	5.42	1/20/06	3.64	1.78	740.58	4/12/06	2.57	2.85	739.51	8/9/06	1.62	3.8	738.56
MW25-10	743.01	6.20	743.01	6.20	1/20/06	3.02	3.18	739.83	4/12/06	1.95	4.25	738.76	8/9/06	1.60	4.6	738.41
MW25-11	740.25	7.00	740.25	7.00	1/20/06	3.70	3.30	736.95	4/12/06	2.55	4.45	735.80	8/9/06	1.95	5.05	735.20
MW25-13	739.64	5.53	739.64	5.53	1/20/06	2.09	3.44	736.20	4/12/06	1.63	3.90	735.74	8/9/06	0.98	4.55	735.09
MW25-15	741.00	7.20	741.00	7.20	1/20/06	4.09	3.11	737.89	4/12/06	3.15	4.05	736.95	8/9/06	2.60	4.6	736.40
MW25-17	743.94	11.27	743.94	11.27	1/20/06	8.02	3.25	740.69	4/12/06	7.07	4.20	739.74	8/9/06	6.92	4.35	739.59
MW25-18	744.35	11.22	744.35	11.22	1/20/06	6.33	4.89	739.46	4/12/06				8/9/06	5.52	5.7	738.65
MW25-19	741.95	12.00	741.95	12.00	1/20/06	8.35	3.65	738.30	4/12/06				8/9/06	6.25	5.75	736.20

Notes:

1. Bedrock wells are not included as part of the LTM program and are not included in this table.

2. Well MW25-3 total depth increased from 9 feet on 8/27/2008 to 9.58 feet on 4/29/2009. Groundwater levels after 8/27/2008 were adjusted to reflect the change in well total depth.

Appendix C
Historic Groundwater Elevations (Events 1 through 13)
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Monitoring Well	Top of Risor Elevation (ft)	Well Depth (ft)	4/29/09 Revised Top of Risor Elevation (ft) [3]	Well Depth (Post-2008) (ft)	Event 3 - June 2007				Event 4 - February 2008				Event 5 - April 2009			
					Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)
MW25-1	743.00	7.77	743.00	7.77	6/4/07	1.27	6.50	736.50	2/26/08	1.88	5.89	737.11	4/27/09	1.68	6.09	736.91
MW25-2	746.36	11.31	746.36	11.31	6/4/07	3.49	7.82	738.54	2/26/08	6.56	4.75	741.61	4/27/09	5.20	6.11	740.25
MW25-3	745.76	9.00	746.34	9.58	6/4/07	0.82	8.18	737.58	2/26/08	4.41	4.59	741.17	4/27/09	3.39	6.19	740.15
MW25-6	744.44	14.27	744.44	14.27	6/4/07	5.72	8.55	735.89	2/26/08	9.73	4.54	739.90	4/27/09	7.84	6.43	738.01
MW25-8	742.46	5.47	742.46	5.47	6/4/07	0.47	5.00	737.46	2/26/08	3.15	2.32	740.14	4/27/09	1.73	3.74	738.72
MW25-9	742.36	5.42	742.36	5.42	6/4/07	0.41	5.01	737.35	2/26/08	3.17	2.25	740.11	4/27/09	1.23	4.19	738.17
MW25-10	743.01	6.20	743.01	6.20	6/4/07		dry		2/26/08	2.46	3.74	739.27	4/27/09	0.29	5.91	737.10
MW25-11	740.25	7.00	740.25	7.00	6/4/07	0.15	6.85	733.40	2/26/08	2.91	4.09	736.16	4/27/09	1.42	5.58	734.67
MW25-13	739.64	5.53	739.64	5.53	6/4/07	0.48	5.05	734.59	2/26/08	1.71	3.82	735.82	4/27/09	0.49	5.04	734.60
MW25-15	741.00	7.20	741.00	7.20	6/4/07		dry		2/26/08	3.77	3.43	737.57	4/27/09	1.75	5.45	735.55
MW25-17	743.94	11.27	743.94	11.27	6/4/07	3.82	7.45	736.49	2/26/08	7.99	3.28	740.66	4/27/09	6.19	5.08	738.86
MW25-18	744.35	11.22	744.35	11.22	6/4/07	4.00	7.22	737.13	2/26/08	11.07	0.15	744.20	4/27/09	5.22	6.00	738.35
MW25-19	741.95	12.00	741.95	12.00	6/4/07	2.97	9.03	732.92	2/26/08	8.00	4.00	737.95	4/27/09	6.50	5.50	736.45

Notes:

1. Bedrock wells are not included as part of the LTM program and are not included in this table.

2. Well MW25-3 total depth increased from 9 feet on 8/27/2008 to 9.58 feet on 4/29/2009. Groundwater levels after 8/27/2008 were adjusted to reflect the change in well total depth.

Appendix C
Historic Groundwater Elevations (Events 1 through 13)
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Monitoring Well	Top of Risor Elevation (ft)	Well Depth (ft)	4/29/09 Revised Top of Risor Elevation (ft) [3]	Well Depth (Post-2008) (ft)	Event 6 - January 2010				Event 7 - August 2010				Event 8 - February 2011			
					Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)
MW25-1	743.00	7.77	743.00	7.77	1/11/10	1.79	5.98	737.02	8/2/10	1.18	6.59	736.41	2/7/11	1.79	5.98	737.02
MW25-2	746.36	11.31	746.36	11.31	1/11/10	5.94	5.37	740.99	8/2/10	4.92	6.39	739.97	2/7/11	4.50	6.81	739.55
MW25-3	745.76	9.00	746.34	9.58	1/11/10	4.44	5.14	741.20	8/2/10	2.00	7.58	738.76	2/7/11	2.70	6.88	739.46
MW25-6	744.44	14.27	744.44	14.27	1/11/10	7.84	6.43	738.01	8/2/10	5.76	8.51	735.93	2/7/11	6.36	7.91	736.53
MW25-8	742.46	5.47	742.46	5.47	1/11/10	2.62	2.85	739.61	8/2/10	0.40	5.07	737.39	2/7/11	0.31	5.16	737.30
MW25-9	742.36	5.42	742.36	5.42	1/11/10	2.92	2.50	739.86	8/2/10	0.44	4.98	737.38	2/7/11	0.83	4.59	737.77
MW25-10	743.01	6.20	743.01	6.20	1/11/10	1.94	4.26	738.75	8/2/10	0.16	6.04	736.97	2/7/11	0.11	6.09	736.92
MW25-11	740.25	7.00	740.25	7.00	1/11/10	1.39	5.61	734.64	8/2/10	0.33	6.67	733.58	(removed during Well Abandonment Fall 2010)			
MW25-13	739.64	5.53	739.64	5.53	1/11/10	0.62	4.91	734.73	8/2/10	0.47	5.06	734.58	2/7/11	0.43	5.10	734.54
MW25-15	741.00	7.20	741.00	7.20	1/11/10	3.02	4.18	736.82	8/2/10	0.30	6.90	734.10	2/7/11	0.63	6.57	734.43
MW25-17	743.94	11.27	743.94	11.27	1/11/10	6.25	5.02	738.92	8/2/10	3.93	7.34	736.60	2/7/11	4.60	6.67	737.27
MW25-18	744.35	11.22	744.35	11.22	1/11/10	5.31	5.91	738.44	8/2/10	4.10	7.12	737.23	2/7/11	4.64	6.58	737.77
MW25-19	741.95	12.00	741.95	12.00	1/11/10	5.79	6.21	735.74	8/2/10	3.21	8.79	733.16	2/7/11	3.89	8.11	733.84

Notes:

1. Bedrock wells are not included as part of the LTM program and are not included in this table.

2. Well MW25-3 total depth increased from 9 feet on 8/27/2008 to 9.58 feet on 4/29/2009. Groundwater levels after 8/27/2008 were adjusted to reflect the change in well total depth.

Appendix C
Historic Groundwater Elevations (Events 1 through 13)
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Monitoring Well	Top of Risor Elevation (ft)	Well Depth (ft)	4/29/09 Revised Top of Risor Elevation (ft) [3]	Well Depth (Post-2008) (ft)	Event 9 - February 2012				Event 10 - May 2013				Event 11 - June 2014			
					Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)
MW25-1	743.00	7.77	743.00	7.77	2/27/12	1.80	7.73	737.07	5/6/13	1.48	6.23	736.77	6/17/14	1.16	6.57	736.43
MW25-2	746.36	11.31	746.36	11.31	2/27/12	6.20	11.26	741.30	5/6/13	5.28	5.97	740.39	6/17/14	4.35	6.91	739.45
MW25-3	745.76	9.00	746.34	9.58	2/27/12	4.86	9.79	741.41	5/6/13	3.64	6.16	740.18	6/17/14	2.01	7.79	738.55
MW25-6	744.44	14.27	744.44	14.27	2/27/12	8.64	14.23	738.85	5/6/13	7.81	6.45	737.99	6/17/14	6.37	7.93	736.51
MW25-8	742.46	5.47	742.46	5.47	2/27/12	2.52	5.41	739.57	5/6/13	1.60	3.83	738.63	6/17/14	0.38	5.04	737.42
MW25-9	742.36	5.42	742.36	5.42	2/27/12	2.59	5.39	739.56	5/6/13	1.48	3.91	738.45	6/17/14	0.45	4.95	737.41
MW25-10	743.01	6.20	743.01	6.20	2/27/12	1.37	6.36	738.02	5/6/13	0.58	5.80	737.21	6/17/14	0.26	6.13	736.88
MW25-11	740.25	7.00	740.25	7.00												
MW25-13	739.64	5.53	739.64	5.53	2/27/12	1.33	4.13	735.51	5/6/13	0.30	5.18	734.46	6/17/14	0.33	5.15	734.49
MW25-15	741.00	7.20	741.00	7.20	2/27/12	2.56	7.19	736.37	5/6/13	1.53	5.65	735.35	6/17/14	0.23	6.97	734.03
MW25-17	743.94	11.27	743.94	11.27	2/27/12	7.14	11.23	739.85	5/6/13	6.36	4.89	739.05	6/17/14	4.48	6.78	737.16
MW25-18	744.35	11.22	744.35	11.22	2/27/12	5.74	11.15	738.94	5/6/13	5.23	5.97	738.38	6/17/14	4.28	6.90	737.45
MW25-19	741.95	12.00	741.95	12.00	2/27/12	6.70	11.98	736.67	5/6/13	6.13	5.87	736.08	6/17/14	3.54	8.46	733.49

Notes:

1. Bedrock wells are not included as part of the LTM program and are not included in this table.

2. Well MW25-3 total depth increased from 9 feet on 8/27/2008 to 9.58 feet on 4/29/2009. Groundwater levels after 8/27/2008 were adjusted to reflect the change in well total depth.

Appendix C
Historic Groundwater Elevations (Events 1 through 13)
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Monitoring Well	Top of Risor Elevation (ft)	Well Depth (ft)	4/29/09 Revised Top of Risor Elevation (ft) [3]	Well Depth (Post-2008) (ft)	Event 12 - March 2015				Event 13 - March 2016				LTM Rounds 1 through 13 Groundwater Elevation (ft) Max/Min Comparison and Range		
					Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Date Measured	Saturated Thickness (ft)	Depth to Groundwater (ft)	Water Level Elevation (ft)	Maximum	Minimum	Range
MW25-1	743.00	7.77	743.00	7.77	3/16/15	2.88	4.83	738.17	3/16/2016	2.37	5.18	737.82	738.17	736.41	1.76
MW25-2	746.36	11.31	746.36	11.31	3/16/15	7.37	3.88	742.48	3/16/2016	7.21	3.84	742.52	742.52	738.54	3.98
MW25-3	745.76	9.00	746.34	9.58	3/16/15	6.71	3.09	743.25	3/16/2016	6.19	3.36	742.98	743.25	737.58	5.67
MW25-6	744.44	14.27	744.44	14.27	3/16/15	11.34	2.93	741.51	3/16/2016	10.80	2.90	741.54	741.54	735.89	5.65
MW25-8	742.46	5.47	742.46	5.47	3/16/15	3.93	1.51	740.95	3/16/2016	3.70	1.50	740.96	740.96	737.30	3.66
MW25-9	742.36	5.42	742.36	5.42	3/16/15	4.07	1.33	741.03	3/16/2016	3.70	1.50	740.86	741.03	737.35	3.68
MW25-10	743.01	6.20	743.01	6.20	3/16/15	5.00	1.38	741.63	3/16/2016	3.93	2.22	740.79	741.63	736.88	4.75
MW25-11	740.25	7.00	740.25	7.00									736.95	733.40	3.55
MW25-13	739.64	5.53	739.64	5.53	3/16/15	2.81	2.66	736.98	3/16/2016	3.12	2.13	737.51	737.51	734.46	3.05
MW25-15	741.00	7.20	741.00	7.20	3/16/15	5.23	1.97	739.03	3/16/2016	4.42	2.53	738.47	739.03	734.03	5.00
MW25-17	743.94	11.27	743.94	11.27	3/16/15	9.52	1.72	742.22	3/16/2016	8.92	1.80	742.14	742.22	736.49	5.73
MW25-18	744.35	11.22	744.35	11.22	3/16/15	7.84	3.32	741.03	3/16/2016	7.30	3.70	740.65	744.20	737.13	7.07
MW25-19	741.95	12.00	741.95	12.00	3/16/15	7.87	4.14	737.81	3/16/2016	8.90	2.90	739.05	739.05	732.92	6.13

Notes:

1. Bedrock wells are not included as part of the LTM program and are not included in this table.

2. Well MW25-3 total depth increased from 9 feet on 8/27/2008 to 9.58 feet on 4/29/2009. Groundwater levels after 8/27/2008 were adjusted to reflect the change in well total depth.

GROUNDWATER ELEVATION REPORT

PARSONS | CLIENT: | DATE: 7/2/10

PROJECT: SEAD-25 LTM | PROJECT NO:

LOCATION: | INSPECTOR: BBO/JK

MONITORING EQUIPMENT: INSTRUMENT	DETECTOR	BGD	TIME	REMARKS	WATER LEVEL INDICATOR: INSTRUMENT	CORRECTION FACTOR

COMMENTS: Collected limited GW levels to determine if GW sampling is possible in future. BBO 7/6

WELL	TIME	TOC DEPTH TO WATER	DEPTH TO PRODUCT	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC. GRAV.	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of: riser, concrete, protective casing, etc.)
MW25-11	1013	6.44						
MW25-13	1015	5.04						
MW25-2	1017	7.19						no well cap
MW25-9	1018	4.95						
MW25-3	1019	7.94						
MW25-8	1021	5.02						well cap on the ground, ants in well

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

GROUNDWATER ELEVATION REPORT

PARSONS | CLIENT: | DATE: 8/2/10

PROJECT: SEAD-25 LTM Round 7

LOCATION:

PROJECT NO:

INSPECTOR: EBO / SD

COMMENTS:

MONITORING EQUIPMENT

INSTRUMENT	DETECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR.

INSTRUMENT	CORRECTION FACTOR

WELL	TIME	Water DEPTH TO WATER	DEPTH TO PRODUCT	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC GRAV	WELL STATUS / COMMENTS (Lock? Well #? Surface Disturbance? Riser marked? Condition: rust, concrete protective casing, etc.)
MW25-1	8:44	6.59						
MW25-11	8:47	6.67						
MW25-13	8:48	5.06						
MW25-15	8:50	6.90						
MW25-19	8:52	8.79						
MW25-6	8:54	8.51						
MW25-17	8:56	7.34						
MW25-18	8:57	7.12						
25-8	8:58	5.07						
25-9	9:00	4.98						
25-10	9:02	6.04						no well cap
25-3	9:03	7.58						
25-2	9:05	6.39						no well cap

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

PAGE OF

GROUNDWATER ELEVATION REPORT

PARSONS | CLIENT: | DATE: 12/20/16

PROJECT: SEAD-25 LTM | PROJECT NO:

LOCATION: SEDA | INSPECTOR: BTO

MONITORING EQUIPMENT:

INSTRUMENT	DETECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR:

INSTRUMENT	CORRECTION FACTOR

COMMENTS: snowy lightly dusty of snow on ground Temp 33°F

WELL	TIME	DEPTH TO WATER	DEPTH TO PRODUCT	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC GRAV	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of riser, concrete, protective casing, etc.)
MW25-1	934	5.99						
25-13	938	4.18'						
25-15	939	4.45'						
25-19	940	5.39						
25-6	941	6.09'						
25-18	942	5.67						
25-17	945	4.69						south of MW25-6, west of MW25-18
25-8	946	2.75						west of MW25-3, PVC lifted?
25-9	948	3.03						
25-10	949	4.81						no well cap
25-3	950	5.11						
25-2	951	5.09						

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

GROUNDWATER ELEVATION REPORT

PARSONS | CLIENT: | DATE: 2/7/2011

PROJECT: SEAD-25 LTM Round 8
LOCATION: Seneca Army Depot
PROJECT NO:
INSPECTOR: BBO / SD

MONITORING EQUIPMENT:

INSTRUMENT	DETECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR:

INSTRUMENT	CORRECTION FACTOR
Pw # 14043	

COMMENTS: ~8 inches of snow over whole site

WELL	TIME	DEPTH TO WATER	DEPTH TO PRODUCT	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC GRAV	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of riser, concrete, protective casing, etc.)
25-1	1539	5.98	7.74					
25-13	1542	5.10	5.48					
25-15	1544	6.57	7.20					
25-9	1546	4.59	5.40					
25-19	1549	8.11	12.0					
25-8	1552	5.16	5.46					
25-6	1554	7.91	14.22					
25-17	1556	6.67	11.30'					
25-10	1558	6.09'	6.37'					
25-2	1600	6.81	11.28'					no well cap
25-3	1601	6.88	9.80					
25-18	1603	6.58	11.18					

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

PAGE OF

GROUNDWATER ELEVATION REPORT

PARSONS	CLIENT:	DATE: 5/6/13
PROJECT:		PROJECT NO:
LOCATION:		INSPECTOR: BBO & SD

MONITORING EQUIPMENT:					WATER LEVEL INDICATOR:		COMMENTS:
INSTRUMENT	DECTECTOR	BGD	TIME	REMARKS	INSTRUMENT	CORRECTION FACTOR	

WELL	TIME	DEPTH TO WATER	DEPTH TO ~~PRODUCT~~ Well Depth	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC. GRAV.	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of: riser, concrete, protective casing, etc.)
25-1	749	6.23	7.71					Locked & bee
-13	755	5.18	5.48					Locked & bees
-15	756	5.65	7.18					Locked & Ants
-19	800	5.87	12.0					Locked &
-6	803	6.45	14.26					Locked
-18	806	5.97	11.20					"
-8	809	3.83	5.43					"
-9	812	3.91	5.39					"
-10	815	5.80	6.38					"
-17	818	4.89	11.25					"
-3	820	6.16	9.80					" & wasp nest
-2	823	5.97	11.25					Locked & bee's, no well cap

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

PAGE OF

GROUNDWATER ELEVATION REPORT

PARSONS | **CLIENT:** | **DATE:** 6/17/14

PROJECT: SEAD-25 LTM Round 11
LOCATION:

PROJECT NO:
INSPECTOR:

MONITORING EQUIPMENT:

INSTRUMENT	DECTECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR:

INSTRUMENT	CORRECTION FACTOR

COMMENTS:

WELL	TIME	DEPTH TO WATER	DEPTH TO ~~PRODUCT~~ Well	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC. GRAV.	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of: riser, concrete, protective casing, etc.)
25-1	1302	6.57	7.73					
25-13	1305	5.15	5.48					lock difficult to open, PVC may have left very difficult to open
25-15	1318	6.97	7.20					
25-19	1328	8.46	12.0					
near old ballfield 25-6	1333	7.93	14.30					
25-17	1337	6.78	11.26					
by road 25-18	1340	6.90	11.18					
25-8	1346	~~4.99~~ 5.04	5.42					ants
25-9	1348	4.95	5.40					
25-10	1352	6.13	6.39					
25-3	1355	7.79	9.80					
25-2	1400	6.91	11.26					

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

GROUNDWATER ELEVATION REPORT

PARSONS | CLIENT: | DATE: 3/16/2015

PROJECT: SEAD-25 LTM Round 12
LOCATION: Romulus, NY
PROJECT NO:
INSPECTOR: BBO / SD

MONITORING EQUIPMENT:

INSTRUMENT	DECTECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR:

INSTRUMENT	CORRECTION FACTOR

COMMENTS: Snow covered ground partially melted

WELL	TIME	DEPTH TO WATER	DEPTH TO ~~PRODUCT~~ Well	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC. GRAV.	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of: riser, concrete, protective casing, etc.)
25-1	1432	4.83	7.71					locked, difficult to open
25-13	1437	2.66	5.47					locked, difficult to open
25-15	1442	1.97	7.20					locked, stiff to open
25-18	1453	3.32	11.16					locked, stiff to open
25-19	1504	4.14	12.01					sprayed lock w/ WD40 to open
25-16 / 25-~~17~~	1508	2.93	14.27					sprayed lock w/ WD40 to open
25-17	1514	1.72	11.24					locked
25-10	1518	1.38	6.38					locked
25-9	1523	1.33	5.40					locked
25-8	1525	1.51	5.44					locked
25-3	1527	3.09	9.80					locked
25-2 / ~~25-3~~	1530	3.88	11.25					locked

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

PAGE 1 OF 1

GROUNDWATER ELEVATION REPORT

PARSONS | **CLIENT:** | **DATE:** 3/16/16

PROJECT: SEAD-25 LTM Rnd 13
LOCATION: ST
PROJECT NO:
INSPECTOR: 320

MONITORING EQUIPMENT:

INSTRUMENT	DETECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR:

INSTRUMENT	CORRECTION FACTOR

COMMENTS: 0.3 ft probe sensor tip length

WELL	TIME	DEPTH TO WATER	DEPTH TO ~~PRODUCT~~ Well	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC. GRAV.	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of: riser, concrete, protective casing, etc.)
25-1	1245	5.48	7.55					wasp nest in well, locked, well hinge cracke
25-13	1257	2.43	5.25					No well cap, locked, wasp nests
25-15	~~1300~~ 1301	2.83	6.95					locked, ants, brush around well
25-19	1307	3.2	11.8					locked, well cap
25-6	1312	3.20	13.7					locked, well cap
~~25-18~~ 25-18	1316	4.0	11.0					locked, well cap
25-17	1319	2.10	10.72					locked, well cap, brush around well
25-9	1323	1.8	5.2					locked, well cap
25-8	1325	1.8	5.2					not locked, PVC lifted, well cap
25-10	1328	2.52	6.15					locked, well cap
25-3	1331	3.66	9.55					locked, PVC lifted, barely opens, well cap
25-2	1337	~~7.14~~ 4.14	11.05					locked, well cap
								locked, well cap

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

APPENDIX D

COMPLETE LTM GROUNDWATER ANALYTICAL DATA (EVENTS 1 THROUGH 13)

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-25 MW25-10 GROUNDWATER 25LM20005 1/31/2006 SA LTM 1 Total		SEAD-25 MW25-10 GROUNDWATER 25LM20015 0-0.1 8/9/2006 SA LTM 2 Total		SEAD-25 MW25-10 GROUNDWATER 25LM20039 0-0.1 3/4/2008 SA LTM 4 Total		SEAD-25 MW25-10 GROUNDWATER 25LM20061 0-0.1 1/13/2010 SA LTM 6 Total		SEAD-25 MW25-10 GROUNDWATER 25LM20083 6.13-6.13 2/9/2011 SA LTM 8 Total		SEAD-25 MW25-10 GROUNDWATER 25LM20094 5.16-5.16 2/28/2012 SA LTM 9 Total		SEAD-25 MW25-10 GROUNDWATER 25LM20105 5.9-5.9 5/7/2013 SA LTM 10 Total		SEAD-25 MW25-10 GROUNDWATER 25LM20117 5.38-5.38 3/17/2015 SA LTM 12 Total		SEAD-25 MW25-10 GROUNDWATER 25LM20123 5.16-5.16 3/17/2016 SA LTM 13 Total		SEAD-25 MW25-13 GROUNDWATER 25LM20006 0-0.1 1/30/2006 SA LTM 1 Total		SEAD-25 MW25-13 GROUNDWATER 25LM20016 0-0.1 8/9/2006 SA LTM 2 Total		GR
Volatile Organic Compounds																																				
1,1,1-Trichloroethane	UG/L	0.62	2%	GA	5	0	2	107	1	U	0.53	J	1	U	0.32	U	0.2	UJ	0.2	U	0.5	UJ	0.37	U	0.37	U	1	U	1	U							
1,1,2,2-Tetrachloroethane	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.09	U	0.38	UJ	0.38	U	0.18	U	0.62	U	0.62	U	1	U	1	U							
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/L	0	0%	GA	5	0	0	102	1	U	1	U	1	U	0.4	U	0.31	U	0.31	UJ	0.5	U	0.36	U	0.36	U	1	UJ	1	U							
1,1,2-Trichloroethane	UG/L	0	0%	GA	1	0	0	107	1	U	1	U	1	U	0.2	U	0.33	UJ	0.33	U	0.13	U	0.33	U	0.33	U	1	U	1	U							
1,1-Dichloroethane	UG/L	3.5	9%	GA	5	0	10	107	1	U	1	U	1	U	0.14	U	0.21	UJ	0.21	U	0.25	U	0.38	U	0.38	U	1	U	1	U							
1,1-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.37	U	0.35	UJ	0.35	U	0.11	U	0.36	U	0.36	U	1	U	1	U							
1,2,4-Trichlorobenzene	UG/L	0	0%	GA	5	0	0	102	1	U	1	U	1	U	0.19	U	0.37	UJ	0.37	U	0.25	U	2.5	U	2.5	U	1	U	1	U							
1,2,4-Trimethylbenzene	UG/L	0.45	7%	GA	5	0	2	30			1	U					0.19	UJ	0.19	U									1	U						
1,2-Dibromo-3-chloropropane	UG/L	0	0%	GA	0.04	0	0	102	1	U	1	U	2	U	0.43	U	0.5	U	0.5	U	0.44	U	1.1	U	1.1	U	1	U	1	U							
1,2-Dibromoethane	UG/L	0	0%	GA	0.0006	0	0	107	1	U	1	U	1	U	0.18	U	0.22	UJ	0.22	U	0.25	U	0.44	U	0.44	U	1	U	1	U							
1,2-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	1	U	1	U	0.4	U	0.15	UJ	0.15	U	0.21	U	0.37	U	0.37	U	1	U	1	U							
1,2-Dichloroethane	UG/L	0.49	3%	GA	0.6	0	3	107	1	U	1	U	1	U	0.14	U	0.2	UJ	0.2	U	0.1	U	0.5	U	0.5	U	1	U	1	U							
1,2-Dichloroethene (total)	UG/L	15	15%	GA	5	2	3	20									0.21	UJ	0.21	UJ																
1,2-Dichloropropane	UG/L	0	0%	GA	1	0	0	107	1	U	1	U	1	U	0.15	U	0.25	UJ	0.25	U	0.13	U	0.67	U	0.67	U	1	U	1	U							
1,3,5-Trimethylbenzene	UG/L	0	0%	GA	5	0	0	30			1	U					0.2	UJ	0.2	U									1	U						
1,3-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	1	U	1	U	0.36	U	0.26	UJ	0.26	U	0.25	U	0.43	U	0.43	U	1	U	1	U							
1,4-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	1	U	1	U	0.34	U	0.24	UJ	0.24	U	0.28	U	0.46	U	0.46	U	1	U	1	U							
Acetone	UG/L	11	7%				7	107	5	U	5	U	10	UJ	5	U	2.2	U	2.2	U	5	U	7	U	7	U	5	U	7.8	UJ							
Benzene	UG/L	62	29%	GA	1	19	31	107	1	U	1	U	1	U	0.18	U	0.26	UJ	0.26	U	0.25	U	0.43	U	0.43	U	1	U	1	U							
Bromodichloromethane	UG/L	0	0%	MCL	80	0	0	107	1	U	1	U	1	U	0.17	U	0.33	UJ	0.33	U	0.25	UJ	0.44	U	0.44	U	1	U	1	U							
Bromoform	UG/L	0	0%	MCL	80	0	0	107	1	U	1	U	1	UJ	0.2	U	0.23	UJ	0.23	U	0.5	U	0.43	U	0.43	UJ	1	U	1	U							
Carbon disulfide	UG/L	0.61	2%				2	107	1	U	1	U	1	U	0.36	U	0.25	U	0.25	U	0.6	U	1	U	1	U	1	U	1	U							
Carbon tetrachloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.36	U	0.22	UJ	0.22	U	0.5	UJ	0.33	U	0.33	U	1	U	1	U							
Chlorobenzene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.26	U	0.22	UJ	0.22	U	0.25	U	0.26	U	0.26	U	1	U	1	U							
Chlorodibromomethane	UG/L	0	0%	MCL	80	0	0	107	1	U	1	U	1	U	0.11	U	0.3	UJ	0.3	U	0.1	U	0.32	U	0.32	U	1	U	1	U							
Chloroethane	UG/L	0.67	2%	GA	5	0	2	107	1	U	1	UJ	2	U	0.21	U	0.55	U	0.55	U	2	U	2.5	U	2.5	U	1	U	1	UJ							
Chloroform	UG/L	0.32	1%	GA	7	0	1	107	1	U	1	U	1	U	0.16	U	0.32	UJ	0.32	U	0.14	U	0.5	U	0.5	U	1	U	1	U							
Cis-1,2-Dichloroethene	UG/L	19	14%	GA	5	4	15	107	1	U	1	U	1	U	0.14	U	0.21	UJ	0.21	U	0.15	U	0.41	U	0.41	U	1	U	1	U							
Cis-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	1	U	1	U	1	U	0.14	U	0.19	UJ	0.19	UJ	0.11	U	0.4	U	0.4	U	1	U	1	U							
Cyclohexane	UG/L	8.6	11%				11	102	1	U	1	U	1	U	0.14	U	0.31	UJ	0.31	U	0.25	U	0.39	U	0.39	U	1	UJ	1	U							
Dichlorodifluoromethane	UG/L	0	0%	GA	5	0	0	82	1	UJ	1	U	1	UJ	0.18	U					0.25	U	0.6	UJ	0.6	U	1	U	1	U							
Diisoproply Ether	UG/L	0	0%				0	20									0.21	U	0.21	U																
Ethyl benzene	UG/L	26	16%	GA	5	12	17	107	1	U	1	U	1	U	0.42	U	0.21	UJ	0.21	U	0.11	U	0.33	U	0.33	U	1	U	1	U							
Isopropylbenzene	UG/L	2.6	9%	GA	5	0	10	107	1	U	1	U	1	U	0.34	U	0.23	UJ	0.23	U	0.1	U	0.35	U	0.35	U	1	U	1	U							
Meta/Para Xylene	UG/L	19	11%	GA	5	4	7	61					1	U	0.81	U	0.59	UJ	0.59	U																
Methyl Acetate	UG/L	0	0%				0	102	1	U	1	U	10	U	0.48	U	0.53	U	0.53	U	0.19	U	1.8	U	1.8	U	1	U	1	U							
Methyl bromide	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	2	U	0.4	U	0.49	U	0.49	UJ	2	U	2.5	UJ	2.5	UJ	1	U	1	U							
Methyl butyl ketone	UG/L	1.9	1%				1	107	5	U	5	U	5	U	0.4	U	1.7	U	1.7	U	1	U	2	U	2	U	5	U	5	U							
Methyl chloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	2	U	0.18	U	0.36	U	0.36	UJ	0.33	U	0.4	U	0.4	U	1	U	1	U							
Methyl cyclohexane	UG/L	4.2	7%				7	102	1	U	1	U	1	U	0.16	U	0.3	U	0.3	U	0.1	U	0.43	U	0.43	U	1	U	1	U							
Methyl ethyl ketone	UG/L	9	10%				11	107	5	U	5	UJ	5	U	1	U	1.3	U	1.3	U	1	U	3.4	U	3.4	U	5	U	5	UJ							
Methyl isobutyl ketone	UG/L	0	0%				0	107	5	U	5	U	5	U	0.34	U	1.3	U	1.3	U	1	U	2.1	U	2.1	U	5	U	5	U							
Methyl Tertbutyl Ether	UG/L	0	0%				0	107	1	U	1	U	1	U	0.13	U	0.36	U	0.36	U	0.2	U	0.3	U	0.3	U	1	U	1	U							
Methylene chloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	UJ	1	U	0.13	U	1.1	U	1.1	U	1	U	2.5	U	2.5	U	1	U	1	UJ							
Naphthalene	UG/L	0.23	4%				1	25									0.3	UJ	0.3	U																
n-Butylbenzene	UG/L	0	0%	GA	5	0	0	20									0.23	UJ	0.23	U																
Ortho Xylene	UG/L	6.4	8%	GA	5	3	5	61					1	U	0.4	U	0.25	UJ	0.25	U																
p-Isopropyltoluene	UG/L	0	0%	GA	5	0	0	10			1	U																	1	U						
Propylbenzene	UG/L	0	0%	GA	5	0	0	10			1	U																	1	U						
sec-Butylbenzene	UG/L	0	0%	GA	5	0	0	20									0.21	UJ	0.21	U																
Styrene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.36	U	0.23	UJ	0.23	U	0.11	U	0.27	U	0.27	U	1	U	1	U							
tert-Butylbenzene	UG/L	0	0%	GA	5	0	0	20									0.31	UJ	0.31	U																
Tetrachloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.42	U	0.4	U	0.4	U	0.15	U	0.74	U	0.74	UJ	1	U	1	U							
Toluene	UG/L	14	11%	GA	5	5	12	107	1	U	1	U	1	U	0.21	U	0.27	UJ	0.27	U	0.33	U	0.48	U	0.48	U	1	U	1	U							
Total Xylenes	UG/L	62	11%	GA	5	3	7	66	3	U	3	U					0.25	UJ	0.25	U	0.2	U	0.23	U	0.23	U	3	U	3	U							
Trans-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.16	U	0.25	U	0.25	U	0.2	U	0.37	U	0.37	U	1	U	1	U							
Trans-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	1	U	1	U	1	U	0.17	U	0.2	UJ	0.2	UJ	0.21	UJ	0.42	U	0.42	U	1	U	1	U							
Trichloroethene	UG/L	2	10%	GA	5	0	11	107	1	U	1	U	1	U	0.19	U	0.28	UJ	0.28	U	0.13	U	0.48	U	0.48	U	1	U	1	U							
Trichlorofluoromethane	UG/L	0	0%	GA	5	0	0	102	1	UJ	1	U	1	U	0.16	U	0.24	U	0.24	U	0.25	U	0.42	U	0.42	U	1	U	1	U							
Vinyl chloride	UG/L	2.6	6%	GA	2	2	6	107	1	U	1	U	1	U	0.22	U	0.25	U	0.25	U	0.18	U	0.5	U	0.5	U	1	U	1	U							

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-10		MW25-10		MW25-10		MW25-10		MW25-10		MW25-10		MW25-10		MW25-10		MW25-13		MW25-13	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20005		25LM20015		25LM20039		25LM20061		25LM20083		25LM20094		25LM20105		25LM20117		25LM20123		25LM20006		25LM20016	
Sample Depth Interval (FT)									0-0.1		0-0.1		0-0.1		0-0.1		6.13-6.13		5.16-5.16		5.9-5.9		5.38-5.38		5.16-5.16		0-0.1		0-0.1	
Sample Date									1/31/2006		8/9/2006		3/4/2008		1/13/2010		2/9/2011		2/28/2012		5/7/2013		3/17/2015		3/17/2016		1/30/2006		8/9/2006	
QC Type									SA		SA		SA		SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									1		2		4		6		8		9		10		12		13		1		2	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Semivolatile Organic Compounds																														
1,1'-Biphenyl	UG/L	0	0%	GA	5	0	0	18	10	U	10	U															9	U		
2,4,5-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18	10	U	10	U															9	U		
2,4,6-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18	10	U	10	U															9	U		
2,4-Dichlorophenol	UG/L	0	0%	GA	5	0	0	18	10	U	10	U															9	U		
2,4-Dimethylphenol	UG/L	0	0%				0	18	10	U	10	U															9	U		
2,4-Dinitrophenol	UG/L	0	0%				0	18	48	U	48	U															47	U		
2,4-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18	10	U	10	U															9	U		
2,6-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18	10	U	10	U															9	U		
2-Chloronaphthalene	UG/L	0	0%				0	18	10	U	10	U															9	U		
2-Chlorophenol	UG/L	0	0%				0	18	10	U	10	U															9	U		
2-Methylnaphthalene	UG/L	0	0%				0	18	10	U	10	U															9	U		
2-Methylphenol	UG/L	0	0%				0	18	10	U	10	U															9	U		
2-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	48	U	48	U															47	U		
2-Nitrophenol	UG/L	0	0%	GA	1	0	0	18	10	U	10	U															9	U		
3,3'-Dichlorobenzidine	UG/L	0	0%	GA	5	0	0	18	19	U	19	U															19	U		
3-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	48	U	48	U															47	U		
4,6-Dinitro-2-methylphenol	UG/L	0	0%	GA	1	0	0	18	48	U	48	U															47	U		
4-Bromophenyl phenyl ether	UG/L	0	0%				0	18	10	U	10	U															9	U		
4-Chloro-3-methylphenol	UG/L	0	0%	GA	1	0	0	18	10	U	10	U															9	U		
4-Chloroaniline	UG/L	0	0%	GA	5	0	0	18	10	U	10	U															9	U		
4-Chlorophenyl phenyl ether	UG/L	0	0%				0	18	10	U	10	U															9	U		
4-Methylphenol	UG/L	0	0%				0	18	10	U	10	U															9	U		
4-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	48	U	48	U															47	U		
4-Nitrophenol	UG/L	0	0%	GA	1	0	0	18	48	U	48	U															47	UJ		
Acenaphthene	UG/L	0.5	6%				1	18	10	U	10	U															9	U		
Acenaphthylene	UG/L	2	22%				4	18	1	J	10	U															9	U		
Acetophenone	UG/L	0	0%				0	18	10	U	10	U															9	U		
Anthracene	UG/L	1	6%				1	18	10	U	10	U															9	U		
Atrazine	UG/L	0	0%	GA	7.5	0	0	18	10	U	10	U															9	U		
Benzaldehyde	UG/L	0	0%				0	18	48	U	48	U															47	U		
Benzo(a)anthracene	UG/L	0	0%				0	18	10	U	10	U															9	U		
Benzo(a)pyrene	UG/L	0	0%	GA	0		0	18	10	U	10	U															9	U		
Benzo(b)fluoranthene	UG/L	0	0%				0	18	10	U	10	U															9	U		
Benzo(ghi)perylene	UG/L	0.6	6%				1	18	10	U	10	U															9	U		
Benzo(k)fluoranthene	UG/L	0	0%				0	18	10	U	10	U															9	U		
Bis(2-Chloroethoxy)methane	UG/L	0	0%	GA	5	0	0	18	10	U	10	U															9	U		
Bis(2-Chloroethyl)ether	UG/L	0	0%	GA	1	0	0	18	10	U	10	U															9	U		
Bis(2-Chloroisopropyl)ether	UG/L	0	0%	GA	5	0	0	18	10	U	10	U															9	U		
Bis(2-Ethylhexyl)phthalate	UG/L	11	6%	GA	5	1	1	18	10	U	10	U															9	U		
Butylbenzylphthalate	UG/L	2	6%				1	18	10	U	10	U															9	U		
Caprolactam	UG/L	0	0%				0	18	10	U	10	U															9	U		
Carbazole	UG/L	0	0%				0	18	10	U	10	U															9	U		
Chrysene	UG/L	0	0%				0	18	10	U	10	U															9	U		
Dibenz(a,h)anthracene	UG/L	0	0%				0	18	10	U	10	U															9	U		
Dibenzofuran	UG/L	0	0%				0	18	10	U	10	U															9	U		
Diethyl phthalate	UG/L	0	0%				0	18	10	U	10	U															9	U		
Dimethylphthalate	UG/L	0	0%				0	18	10	U	10	U															9	U		
Di-n-butylphthalate	UG/L	0	0%	GA	50	0	0	18	10	U	10	U															9	U		
Di-n-octylphthalate	UG/L	0	0%				0	18	10	U	10	U															9	U		
Fluoranthene	UG/L	0	0%				0	18	10	U	10	U															9	U		
Fluorene	UG/L	0	0%				0	18	10	U	10	UJ															9	U		
Hexachlorobenzene	UG/L	0	0%	GA	0.04	0	0	18	10	U	10	U															9	U		
Hexachlorobutadiene	UG/L	0	0%	GA	0.5	0	0	18	10	U	10	U															9	U		
Hexachlorocyclopentadiene	UG/L	0	0%	GA	5	0	0	18	43	U	43	U															42	U		
Hexachloroethane	UG/L	0	0%	GA	5	0	0	18	10	U	10	U															9	U		
Indeno(1,2,3-cd)pyrene	UG/L	0	0%				0	18	10	U	10	U															9	U		
Isophorone	UG/L	0	0%				0	18	10	U	10	U															9	U		
Naphthalene	UG/L	2	6%				1	18	10	U	10	U															9	U		
Nitrobenzene	UG/L	0	0%	GA	0.4	0	0	18	10	U	10	U															9	U		
N-Nitroso-di-n-propylamine	UG/L	0	0%				0	18	10	U	10	U															9	U		
N-Nitrosodiphenylamine	UG/L	0	0%				0	18	10	U	10	U															9	U		
Pentachlorophenol	UG/L	0	0%	GA	1	0	0	18	48	U	48	U															47	U		
Phenanthrene	UG/L	0	0%				0	18	10	U	10	U															9	U		
Phenol	UG/L	0	0%	GA	1	0	0	18	10	U	10	U															9	U		
Pyrene	UG/L	0	0%				0	18	10	U	10	U															9	U		

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-10		MW25-10		MW25-10		MW25-10		MW25-10		MW25-10		MW25-10		MW25-10		MW25-13		MW25-13	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20005		25LM20015		25LM20039		25LM20061		25LM20083		25LM20094		25LM20105		25LM20117		25LM20123		25LM20006		25LM20016	
Sample Depth Interval (FT)									0-0.1		0-0.1		0-0.1		0-0.1		6.13-6.13		5.16-5.16		5.9-5.9		5.38-5.38		5.16-5.16		0-0.1		0-0.1	
Sample Date									1/31/2006		8/9/2006		3/4/2008		1/13/2010		2/9/2011		2/28/2012		5/7/2013		3/17/2015		3/17/2016		1/30/2006		8/9/2006	
QC Type									SA		SA		SA		SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									1		2		4		6		8		9		10		12		13		1		2	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																														
Iron	UG/L	15,700	85%	GA	300	56	84	99	62.8	J	**358**		100	U	**508**				231	J	200	J	200		25	J	**320**	**J**		
Sodium	UG/L	58,100	100%	GA	20,000	14	99	99	8,870		6,530	J	6,090		6,420				5,040		4,800	J	9,100		5,000		**40,600**			
Wet Chemistry - MEE																														
Chloride	MG/L	97.9	78%	GA	250	0	78	100	0.73		0.71	J	0.2	U	2.1				0.45	J	1	U	2.1		0.73		2.5			
Ethane	UG/L	1.1	5%				5	104	2	U	2	U	1	U	0.21	U			0.58	U	0.81	U	0.55	U	0.55	U	2	U		
Ethene	UG/L	4.6	5%				5	104	2	U	2	U	1	U	0.22	U			0.69	U	0.73	U	0.5	U	0.5	U	2	U		
Methane	UG/L	170	57%				59	104	2	U	2	U	2	U	0.14	U			1.2	J	0.45	U	0.54	J	0.29	U	2	U		
Nitrate	MG/L	6.4	68%	GA	10	0	45	66							0.05	UJ			0.02	J	0.026	J	0.066		0.082					
Nitrate Nitrogen	MG/L	1	45%				13	29	0.05	U	0.05	U	0.102	J													0.05	U		
Nitrate/Nitrite Nitrogen	MG/L	1	64%	GA	10	0	16	25					0.102		0.003	UJ														
Nitrite	MG/L	0.73	27%	GA	1	0	18	66							0.007	UJ			0.015	J	0.01	U	0.01	U	0.01	U				
Nitrite Nitrogen	MG/L	0.087	3%				1	29	0.05	U	0.05	U	0.01	UJ													0.05	U		
Sulfate	MG/L	182	100%	GA	250	0	100	100	18.1		18.4		12.9		27.1	J			14	J	14	J	4.2		8.5		15.6			
Field Measurement - Hach Kit																														
Conductivity	S/m	1.26	100%				90	90	0.464		0.701		0.473		0.396								0.365		0.389		0.492		0.699	
Conductivity (post)	S/m	0.844	100%				3	3																						
Conductivity (pre)	S/m	0.83	100%				3	3																						
Dissolved Oxygen	MG/L	12.6	100%				86	86	4.22		4.23		3.65										12.6		8.36		0.94		4.1	
Dissolved Oxygen (post)	MG/L	5.17	100%				3	3																						
Dissolved Oxygen (pre)	MG/L	5.36	100%				3	3																						
Nitrate Nitrogen	MG/L	0.5	100%				3	3																						
Nitrite Nitrogen	MG/L	0.5	100%				3	3																						
ORP	mV	259	100%				90	90	107		138.8		130		230								165		175		38		-22.2	
ORP (post)	mV	197	100%				3	3																						
ORP (pre)	mV	193	100%				3	3																						
pH	Std units	8.37	100%				90	90	6.97		6.56		7.31		7.19								7.64		7.99		7.27		6.98	
pH (post)	Std units	7.38	100%				3	3																						
pH (pre)	Std units	7.38	100%				3	3																						
Sulfide	MG/L	1.04	89%				72	81	0.1		0.28		0.02		0.09						0.01		0.01		0.01		0.02			
Temperature	DEG C	26.55	100%				93	93	5		21.56		3.6		5.6								2.7		5.7		3.8		23.42	
Turbidity	NTU	195	100%				90	90	1.09		195		2.36		3.3								3.66		1.13		21		100	
Turbidity (post)	NTU	7.6	100%				2	2																						
Turbidity (pre)	NTU	5.7	100%				1	1																						

Notes:
1. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
2. Shading indicates concentration above cleanup goal.

U = compound was not detected
J = the reported value is an estimated concentration
UJ = the compound was not detected; the associated reporting limit is approximate
ND = Non-detect

SA = Sample
DU = Duplicate

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-13		MW25-13		MW25-15		MW25-15		MW25-15		MW25-15		MW25-15		MW25-15		MW25-15		MW25-17	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20040		25LM20095		25LM20007		25LM20017		25LM20041		25LM20052		25LM20063		25LM20085		25LM20096		25LM20107		25LM20008	
Sample Depth Interval (FT)									0-0.1		4.32-4.32		0-0.1		0-0.1		0-0.1		0-0.1		6.53-6.53		4.8-4.8		6.1-6.1		0-0.1	
Sample Date									3/3/2008		2/28/2012		1/31/2006		8/14/2006		3/3/2008		4/29/2009		1/13/2010		2/9/2011		2/28/2012		5/7/2013		1/30/2006	
QC Type									SA		SA		SA		SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									4		9		1		2		4		5		6		8		9		10		1	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Volatile Organic Compounds																														
1,1,1-Trichloroethane	UG/L	0.62	2%	GA	5	0	2	107	1	U	0.2	U	1	U	1	U	1	U	1	U	0.32	U	0.2	UJ	0.2	U	0.5	UJ	1	U
1,1,2,2-Tetrachloroethane	UG/L	0	0%	GA	5	0	0	107	1	U	0.38	U	1	U	1	U	1	U	1	U	0.09	U	0.38	UJ	0.38	U	0.18	U	1	U
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/L	0	0%	GA	5	0	0	102	1	U	0.31	UJ	1	U	1	U	1	U	1	U	0.4	U	0.31	U	0.31	UJ	0.5	U	1	UJ
1,1,2-Trichloroethane	UG/L	0	0%	GA	1	0	0	107	1	U	0.33	U	1	U	1	U	1	U	1	U	0.2	U	0.33	UJ	0.33	U	0.13	U	1	U
1,1-Dichloroethane	UG/L	3.5	9%	GA	5	0	10	107	1	U	0.21	U	1	U	1	U	1	U	1	U	0.14	U	0.21	UJ	0.21	U	0.25	U	1	U
1,1-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	0.35	U	1	U	1	U	1	U	1	U	0.37	U	0.35	UJ	0.35	U	0.11	U	1	U
1,2,4-Trichlorobenzene	UG/L	0	0%	GA	5	0	0	102	1	U	0.37	U	1	U	1	U	1	U	1	U	0.19	U	0.37	UJ	0.37	U	0.25	U	1	U
1,2,4-Trimethylbenzene	UG/L	0.45	7%	GA	5	0	2	30			0.19	U			1	U							0.19	UJ	0.19	U				
1,2-Dibromo-3-chloropropane	UG/L	0	0%	GA	0.04	0	0	102	2	U	0.5	U	1	U	1	U	2	U	2	U	0.43	U	0.5	U	0.5	U	0.44	U	1	U
1,2-Dibromoethane	UG/L	0	0%	GA	0.0006	0	0	107	1	U	0.22	U	1	U	1	U	1	U	1	U	0.18	U	0.22	UJ	0.22	U	0.25	U	1	U
1,2-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	0.15	U	1	U	1	U	1	U	1	U	0.4	U	0.15	UJ	0.15	U	0.21	U	1	U
1,2-Dichloroethane	UG/L	0.49	3%	GA	0.6	0	3	107	1	U	0.2	U	1	U	1	U	1	U	1	U	0.14	U	0.2	UJ	0.2	U	0.1	U	1	U
1,2-Dichloroethene (total)	UG/L	15	15%	GA	5	2	3	20			0.21	UJ											0.21	UJ	0.21	UJ				
1,2-Dichloropropane	UG/L	0	0%	GA	1	0	0	107	1	U	0.25	U	1	U	1	U	1	U	1	U	0.15	U	0.25	UJ	0.25	U	0.13	U	1	U
1,3,5-Trimethylbenzene	UG/L	0	0%	GA	5	0	0	30			0.2	U			1	U							0.2	UJ	0.2	U				
1,3-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	0.26	U	1	U	1	U	1	U	1	U	0.36	U	0.26	UJ	0.26	U	0.25	U	1	U
1,4-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	0.24	U	1	U	1	U	1	U	1	U	0.34	U	0.24	UJ	0.24	U	0.28	U	1	U
Acetone	UG/L	11	7%				7	107	10	UJ	2.2	U	5	U	12	UJ	10	UJ	5	U	5	U	2.2	U	2.2	U	5	U	5	U
Benzene	UG/L	62	29%	GA	1	19	31	107	1	U	0.26	U	1	U	1	U	1	U	1	U	0.18	U	0.26	UJ	0.26	U	0.25	U	1	U
Bromodichloromethane	UG/L	0	0%	MCL	80	0	0	107	1	U	0.33	U	1	U	1	U	1	U	1	U	0.17	U	0.33	UJ	0.33	U	0.25	UJ	1	U
Bromoform	UG/L	0	0%	MCL	80	0	0	107	1	UJ	0.23	U	1	U	1	U	1	UJ	1.2	U	0.2	U	0.23	UJ	0.23	U	0.5	U	1	U
Carbon disulfide	UG/L	0.61	2%				2	107	1	U	0.25	U	1	U	1	U	1	U	1	U	0.36	U	0.25	U	0.25	U	0.6	U	1	U
Carbon tetrachloride	UG/L	0	0%	GA	5	0	0	107	1	U	0.22	U	1	U	1	U	1	U	1	U	0.36	U	0.22	UJ	0.22	U	0.5	UJ	1	U
Chlorobenzene	UG/L	0	0%	GA	5	0	0	107	1	U	0.22	U	1	U	1	U	1	U	1	U	0.26	U	0.22	UJ	0.22	U	0.25	U	1	U
Chlorodibromomethane	UG/L	0	0%	MCL	80	0	0	107	1	U	0.3	U	1	U	1	U	1	U	1	U	0.11	U	0.3	UJ	0.3	U	0.1	U	1	U
Chloroethane	UG/L	0.67	2%	GA	5	0	2	107	2	U	0.55	U	1	U	1	U	2	U	1	U	0.21	U	0.55	U	0.55	U	2	U	1	U
Chloroform	UG/L	0.32	1%	GA	7	0	1	107	1	U	0.32	U	1	U	1	U	1	U	1	U	0.16	U	0.32	UJ	0.32	U	0.14	U	1	U
Cis-1,2-Dichloroethene	UG/L	19	14%	GA	5	4	15	107	1	U	0.21	U	1	U	1	U	1	U	1	U	0.14	U	0.21	UJ	0.21	U	0.15	U	1	U
Cis-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	1	U	0.19	UJ	1	U	1	U	1	U	1	U	0.14	U	0.19	UJ	0.19	UJ	0.11	U	1	U
Cyclohexane	UG/L	8.6	11%				11	102	1	U	0.31	U	1	U	1	U	1	U	1	U	0.14	U	0.31	UJ	0.31	U	0.25	U	1	UJ
Dichlorodifluoromethane	UG/L	0	0%	GA	5	0	0	82	1	UJ			1	UJ	1	U	1	UJ	1	U	0.18	U					0.25	U	1	U
Diisoproply Ether	UG/L	0	0%				0	20			0.21	U											0.21	U	0.21	U				
Ethyl benzene	UG/L	26	16%	GA	5	12	17	107	1	U	0.21	U	1	U	1	U	1	U	1	U	0.42	U	0.21	UJ	0.21	U	0.11	U	1	U
Isopropylbenzene	UG/L	2.6	9%	GA	5	0	10	107	1	U	0.23	U	1	U	1	U	1	U	1	U	0.34	U	0.23	UJ	0.23	U	0.1	U	1	U
Meta/Para Xylene	UG/L	19	11%	GA	5	4	7	61	1	U	0.59	U					1	U	2	U	0.81	U	0.59	UJ	0.59	U				
Methyl Acetate	UG/L	0	0%				0	102	10	U	0.53	U	1	U	1	U	10	U	2	U	0.48	U	0.53	U	0.53	U	0.19	U	1	U
Methyl bromide	UG/L	0	0%	GA	5	0	0	107	2	U	0.49	UJ	1	U	1	UJ	2	U	1	UJ	0.4	U	0.49	U	0.49	UJ	2	U	1	U
Methyl butyl ketone	UG/L	1.9	1%				1	107	5	U	1.7	U	5	U	5	U	5	U	5	U	0.4	U	1.7	U	1.7	U	1	U	5	U
Methyl chloride	UG/L	0	0%	GA	5	0	0	107	2	U	0.36	UJ	1	U	1	U	2	U	1	U	0.18	U	0.36	U	0.36	UJ	0.33	U	1	U
Methyl cyclohexane	UG/L	4.2	7%				7	102	1	U	0.3	U	1	U	1	U	1	U	1	U	0.16	U	0.3	U	0.3	U	0.1	U	1	U
Methyl ethyl ketone	UG/L	9	10%				11	107	5	U	1.3	U	5	U	5	U	5	U	5	U	1	U	1.3	U	1.3	U	1	U	5	U
Methyl isobutyl ketone	UG/L	0	0%				0	107	5	U	1.3	U	5	U	5	U	5	U	5	U	0.34	U	1.3	U	1.3	U	1	U	5	U
Methyl Tertbutyl Ether	UG/L	0	0%				0	107	1	U	0.36	U	1	U	1	U	1	U	1	U	0.13	U	0.36	U	0.36	U	0.2	U	1	U
Methylene chloride	UG/L	0	0%	GA	5	0	0	107	1	U	1.1	U	1	U	1	U	1	U	1	U	0.13	U	1.1	U	1.1	U	1	U	1	U
Naphthalene	UG/L	0.23	4%				1	25			0.3	U											0.3	UJ	0.3	U				
n-Butylbenzene	UG/L	0	0%	GA	5	0	0	20			0.23	U											0.23	UJ	0.23	U				
Ortho Xylene	UG/L	6.4	8%	GA	5	3	5	61	1	U	0.25	U					1	U	1	U	0.4	U	0.25	UJ	0.25	U				
p-Isopropyltoluene	UG/L	0	0%	GA	5	0	0	10							1	U														
Propylbenzene	UG/L	0	0%	GA	5	0	0	10							1	U														
sec-Butylbenzene	UG/L	0	0%	GA	5	0	0	20			0.21	U											0.21	UJ	0.21	U				
Styrene	UG/L	0	0%	GA	5	0	0	107	1	U	0.23	U	1	U	1	U	1	U	1	U	0.36	U	0.23	UJ	0.23	U	0.11	U	1	U
tert-Butylbenzene	UG/L	0	0%	GA	5	0	0	20			0.31	U											0.31	UJ	0.31	U				
Tetrachloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	0.4	U	1	U	1	U	1	U	1	U	0.42	U	0.4	U	0.4	U	0.15	U	1	U
Toluene	UG/L	14	11%	GA	5	5	12	107	1	U	0.27	U	1	U	1	U	1	U	1	U	0.21	U	0.27	UJ	0.27	U	0.33	U	1	U
Total Xylenes	UG/L	62	11%	GA	5	3	7	66			0.25	U	3	U	3	U							0.25	UJ	0.25	U	0.2	U	3	U
Trans-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	0.25	U	1	U	1	U	1	U	1	U	0.16	U	0.25	U	0.25	U	0.2	U	1	U
Trans-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	1	U	0.2	UJ	1	U	1	U	1	U	1	U	0.17	U	0.2	UJ	0.2	UJ	0.21	UJ	1	U
Trichloroethene	UG/L	2	10%	GA	5	0	11	107	1	U	0.28	U	1	U	1	U	1	U	1	U	0.19	U	0.28	UJ	0.28	U	0.13	U	1	U
Trichlorofluoromethane	UG/L	0	0%	GA	5	0	0	102	1	U	0.24	U	1	UJ	1	U	1	U	1	U	0.16	U	0.24	U	0.24	U	0.25	U	1	U
Vinyl chloride	UG/L	2.6	6%	GA	2	2	6	107	1	U	0.25	U	1	U	1	U	1	U	1	U	0.22	U	0.25	U	0.25	U	0.18	U	1	U

Area									SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25
Loc ID									MW25-13	MW25-13	MW25-15	MW25-15	MW25-15	MW25-15	MW25-15	MW25-15	MW25-15	MW25-15	MW25-17
Matrix									GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER
Sample ID									25LM20040	25LM20095	25LM20007	25LM20017	25LM20041	25LM20052	25LM20063	25LM20085	25LM20096	25LM20107	25LM20008
Sample Depth Interval (FT)									0-0.1	4.32-4.32	0-0.1	0-0.1	0-0.1	0-0.1	0-0.1	6.53-6.53	4.8-4.8	6.1-6.1	0-0.1
Sample Date									3/3/2008	2/28/2012	1/31/2006	8/14/2006	3/3/2008	4/29/2009	1/13/2010	2/9/2011	2/28/2012	5/7/2013	1/30/2006
QC Type									SA	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA
Study ID									LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM
Sample Round									4	9	1	2	4	5	6	8	9	10	1
Filtered									Total	Total	Total	Total	Total	Total	Total	Total	Total	Total	Total
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual
Semivolatile Organic Compounds																			
1,1'-Biphenyl	UG/L	0	0%	GA	5	0	0	18			9 U								9 U
2,4,5-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18			9 U								9 U
2,4,6-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18			9 U								9 U
2,4-Dichlorophenol	UG/L	0	0%	GA	5	0	0	18			9 U								9 U
2,4-Dimethylphenol	UG/L	0	0%				0	18			9 U								9 U
2,4-Dinitrophenol	UG/L	0	0%				0	18			47 U								47 U
2,4-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18			9 U								9 U
2,6-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18			9 U								9 U
2-Chloronaphthalene	UG/L	0	0%				0	18			9 U								9 U
2-Chlorophenol	UG/L	0	0%				0	18			9 U								9 U
2-Methylnaphthalene	UG/L	0	0%				0	18			9 U								9 U
2-Methylphenol	UG/L	0	0%				0	18			9 U								9 U
2-Nitroaniline	UG/L	0	0%	GA	5	0	0	18			47 U								47 U
2-Nitrophenol	UG/L	0	0%	GA	1	0	0	18			9 U								9 U
3,3'-Dichlorobenzidine	UG/L	0	0%	GA	5	0	0	18			19 U								19 U
3-Nitroaniline	UG/L	0	0%	GA	5	0	0	18			47 U								47 U
4,6-Dinitro-2-methylphenol	UG/L	0	0%	GA	1	0	0	18			47 U								47 U
4-Bromophenyl phenyl ether	UG/L	0	0%				0	18			9 U								9 U
4-Chloro-3-methylphenol	UG/L	0	0%	GA	1	0	0	18			9 U								9 U
4-Chloroaniline	UG/L	0	0%	GA	5	0	0	18			9 U								9 U
4-Chlorophenyl phenyl ether	UG/L	0	0%				0	18			9 U								9 U
4-Methylphenol	UG/L	0	0%				0	18			9 U								9 U
4-Nitroaniline	UG/L	0	0%	GA	5	0	0	18			47 U								47 U
4-Nitrophenol	UG/L	0	0%	GA	1	0	0	18			47 U								47 UJ
Acenaphthene	UG/L	0.5	6%				1	18			9 U								9 U
Acenaphthylene	UG/L	2	22%				4	18			0.7 J								9 U
Acetophenone	UG/L	0	0%				0	18			9 U								9 U
Anthracene	UG/L	1	6%				1	18			9 U								9 U
Atrazine	UG/L	0	0%	GA	7.5	0	0	18			9 U								9 U
Benzaldehyde	UG/L	0	0%				0	18			47 U								47 U
Benzo(a)anthracene	UG/L	0	0%				0	18			9 U								9 U
Benzo(a)pyrene	UG/L	0	0%	GA	0		0	18			9 U								9 U
Benzo(b)fluoranthene	UG/L	0	0%				0	18			9 U								9 U
Benzo(ghi)perylene	UG/L	0.6	6%				1	18			9 U								9 U
Benzo(k)fluoranthene	UG/L	0	0%				0	18			9 U								9 U
Bis(2-Chloroethoxy)methane	UG/L	0	0%	GA	5	0	0	18			9 U								9 U
Bis(2-Chloroethyl)ether	UG/L	0	0%	GA	1	0	0	18			9 U								9 U
Bis(2-Chloroisopropyl)ether	UG/L	0	0%	GA	5	0	0	18			9 U								9 U
Bis(2-Ethylhexyl)phthalate	UG/L	11	6%	GA	5	1	1	18			9 U								9 U
Butylbenzylphthalate	UG/L	2	6%				1	18			9 U								9 U
Caprolactam	UG/L	0	0%				0	18			9 U								9 U
Carbazole	UG/L	0	0%				0	18			9 U								9 U
Chrysene	UG/L	0	0%				0	18			9 U								9 U
Dibenz(a,h)anthracene	UG/L	0	0%				0	18			9 U								9 U
Dibenzofuran	UG/L	0	0%				0	18			9 U								9 U
Diethyl phthalate	UG/L	0	0%				0	18			9 U								9 U
Dimethylphthalate	UG/L	0	0%				0	18			9 U								9 U
Di-n-butylphthalate	UG/L	0	0%	GA	50	0	0	18			9 U								9 U
Di-n-octylphthalate	UG/L	0	0%				0	18			9 U								9 U
Fluoranthene	UG/L	0	0%				0	18			9 U								9 U
Fluorene	UG/L	0	0%				0	18			9 U								9 U
Hexachlorobenzene	UG/L	0	0%	GA	0.04	0	0	18			9 U								9 U
Hexachlorobutadiene	UG/L	0	0%	GA	0.5	0	0	18			9 U								9 U
Hexachlorocyclopentadiene	UG/L	0	0%	GA	5	0	0	18			42 U								42 U
Hexachloroethane	UG/L	0	0%	GA	5	0	0	18			9 U								9 U
Indeno(1,2,3-cd)pyrene	UG/L	0	0%				0	18			9 U								9 U
Isophorone	UG/L	0	0%				0	18			9 U								9 U
Naphthalene	UG/L	2	6%				1	18			9 U								9 U
Nitrobenzene	UG/L	0	0%	GA	0.4	0	0	18			9 U								9 U
N-Nitroso-di-n-propylamine	UG/L	0	0%				0	18			9 U								9 U
N-Nitrosodiphenylamine	UG/L	0	0%				0	18			9 U								9 U
Pentachlorophenol	UG/L	0	0%	GA	1	0	0	18			47 U								47 U
Phenanthrene	UG/L	0	0%				0	18			9 U								9 U
Phenol	UG/L	0	0%	GA	1	0	0	18			9 U								9 U
Pyrene	UG/L	0	0%				0	18			9 U								9 U

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-13		MW25-13		MW25-15		MW25-15		MW25-15		MW25-15		MW25-15		MW25-15		MW25-15		MW25-17	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20040		25LM20095		25LM20007		25LM20017		25LM20041		25LM20052		25LM20063		25LM20085		25LM20096		25LM20107		25LM20008	
Sample Depth Interval (FT)									0-0.1		4.32-4.32		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		6.53-6.53		4.8-4.8		6.1-6.1		0-0.1	
Sample Date									3/3/2008		2/28/2012		1/31/2006		8/14/2006		3/3/2008		4/29/2009		1/13/2010		2/9/2011		2/28/2012		5/7/2013		1/30/2006	
QC Type									SA		SA		SA		SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									4		9		1		2		4		5		6		8		9		10		1	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																														
Iron	UG/L	15,700	85%	GA	300	56	84	99			**2,320**	**J**	56	J	**850**		100	U	30	J	**769**				**3,840**	**J**	**530**		46.1	
Sodium	UG/L	58,100	100%	GA	20,000	14	99	99			16,100		3,080		6,630	J	6,340		3,500		3,620				3,130		2,800		4,240	
Wet Chemistry - MEE																														
Chloride	MG/L	97.9	78%	GA	250	0	78	100			0.54	J	0.66		1.4	J	0.2	U	0.2	U	0.5	U			0.56	J	1	U	0.7	
Ethane	UG/L	1.1	5%				5	104			0.58	U	2	U	2	U	1	U	1	U	0.16	U	0.58	U	0.58	U	0.81	U	2	U
Ethene	UG/L	4.6	5%				5	104			0.69	U	2	U	2	U	1	U	1	U	0.17	U	0.69	U	0.69	U	0.73	U	2	U
Methane	UG/L	170	57%				59	104			1.2	J	2	U	2	U	2	U	2	U	0.14	U	2.1	J	1.2	J	0.45	U	2	U
Nitrate	MG/L	6.4	68%	GA	10	0	45	66			0.051								0.05	U	0.05	UJ			0.018	J	0.019	J		
Nitrate Nitrogen	MG/L	1	45%				13	29					0.05	U	0.05	U	0.16	J											0.05	U
Nitrate/Nitrite Nitrogen	MG/L	1	64%	GA	10	0	16	25									0.16				0.003	UJ								
Nitrite	MG/L	0.73	27%	GA	1	0	18	66			0.015	J							0.01	U	0.007	UJ			0.02	J	0.01	U		
Nitrite Nitrogen	MG/L	0.087	3%				1	29					0.05	U	0.087		0.01	UJ											0.05	U
Sulfate	MG/L	182	100%	GA	250	0	100	100			18	J	14.4		17.9		13.3		20.3		24.8	J			14	J	9.5		17.2	
Field Measurement - Hach Kit																														
Conductivity	S/m	1.26	100%				90	90	0.639				0.36		0.651		0.477				0.38				0.419				0.462	
Conductivity (post)	S/m	0.844	100%				3	3																						
Conductivity (pre)	S/m	0.83	100%				3	3																						
Dissolved Oxygen	MG/L	12.6	100%				86	86	4.79				2.93		1.99		4.57								1.55				8.46	
Dissolved Oxygen (post)	MG/L	5.17	100%				3	3																						
Dissolved Oxygen (pre)	MG/L	5.36	100%				3	3																						
Nitrate Nitrogen	MG/L	0.5	100%				3	3																						
Nitrite Nitrogen	MG/L	0.5	100%				3	3																						
ORP	mV	259	100%				90	90	97				82		222.1		139				213				97				68	
ORP (post)	mV	197	100%				3	3																						
ORP (pre)	mV	193	100%				3	3																						
pH	Std units	8.37	100%				90	90	7.52				7.2		5.8		7.25				7.23				7				7.69	
pH (post)	Std units	7.38	100%				3	3																						
pH (pre)	Std units	7.38	100%				3	3																						
Sulfide	MG/L	1.04	89%				72	81	0.01	U			0.01	U	0.8		0.01	U	0.01	U	0.17				0.05		0.05		0.01	
Temperature	DEG C	26.55	100%				93	93	3				5.3		18.76		4.7				6.1				5.1				6.3	
Turbidity	NTU	195	100%				90	90	16.4				1.1		27.4		3.58				1.5				4.2				3.4	
Turbidity (post)	NTU	7.6	100%				2	2																						
Turbidity (pre)	NTU	5.7	100%				1	1																						

Notes:
1. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
2. Shading indicates concentration above cleanup goal.

U = compound was not detected
J = the reported value is an estimated concentration
UJ = the compound was not detected; the associated reporting limit is approximate
ND = Non-detect

SA = Sample
DU = Duplicate

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		MW25-17	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20018		25LM20024		25LM20028		25LM20032		25LM20033		25LM20043		25LM20055		25LM20065		25LM20076		25LM20088		25LM20098	
Sample Depth Interval (FT)									0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0		10.3-10.3		10.23-10.23		10.25-10.25	
Sample Date									8/11/2006		6/7/2007		6/7/2007		3/4/2008		3/4/2008		4/28/2009		1/14/2010		8/5/2010		2/10/2011		2/28/2012		5/8/2013	
QC Type									SA		DU		SA		DU		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									2		3		3		4		4		5		6		7		8		9		10	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Volatile Organic Compounds																														
1,1,1-Trichloroethane	UG/L	0.62	2%	GA	5	0	2	107	1	U	1	U	1	U	1	U	1	U	1	U	0.32	U	0.32	U	0.2	UJ	0.2	U	0.5	U
1,1,2,2-Tetrachloroethane	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.09	U	0.09	U	0.38	UJ	0.38	U	0.18	U
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/L	0	0%	GA	5	0	0	102	1	U					1	U	1	U	1	U	0.4	U	0.4	U	0.31	U	0.31	UJ	0.5	U
1,1,2-Trichloroethane	UG/L	0	0%	GA	1	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.2	U	0.2	U	0.33	UJ	0.33	U	0.13	U
1,1-Dichloroethane	UG/L	3.5	9%	GA	5	0	10	107	1	U	1	U	1	U	1	U	1	U	1	U	0.14	U	0.14	U	0.21	UJ	0.21	U	0.25	U
1,1-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.37	U	0.37	U	0.35	UJ	0.35	U	0.11	U
1,2,4-Trichlorobenzene	UG/L	0	0%	GA	5	0	0	102	1	U					1	U	1	U	1	U	0.19	U	0.19	U	0.37	UJ	0.37	U	0.25	UJ
1,2,4-Trimethylbenzene	UG/L	0.45	7%	GA	5	0	2	30	1	U															0.19	UJ	0.19	U		
1,2-Dibromo-3-chloropropane	UG/L	0	0%	GA	0.04	0	0	102	1	U					2	U	2	U	2	U	0.43	U	0.43	U	0.5	U	0.5	U	0.44	U
1,2-Dibromoethane	UG/L	0	0%	GA	0.0006	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.18	U	0.18	U	0.22	UJ	0.22	U	0.25	U
1,2-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U					1	U	1	U	1	U	0.4	U	0.4	U	0.15	UJ	0.15	U	0.21	U
1,2-Dichloroethane	UG/L	0.49	3%	GA	0.6	0	3	107	1	U	1	U	1	U	1	U	1	U	1	U	0.14	U	0.14	U	0.2	UJ	0.2	U	0.1	U
1,2-Dichloroethene (total)	UG/L	15	15%	GA	5	2	3	20																	0.21	UJ	0.21	UJ		
1,2-Dichloropropane	UG/L	0	0%	GA	1	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.15	U	0.15	U	0.25	UJ	0.25	U	0.13	U
1,3,5-Trimethylbenzene	UG/L	0	0%	GA	5	0	0	30	1	U															0.2	UJ	0.2	U		
1,3-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U					1	U	1	U	1	U	0.36	U	0.36	U	0.26	UJ	0.26	U	0.25	U
1,4-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U					1	U	1	U	1	U	0.34	U	0.34	U	0.24	UJ	0.24	U	0.28	U
Acetone	UG/L	11	7%				7	107	5	U	5	U	5	U	10	UJ	10	UJ	5	U	5	U	1.6	U	2.2	U	2.2	U	5	U
Benzene	UG/L	62	29%	GA	1	19	31	107	1	U	1	U	1	U	1	U	1	U	1	U	0.18	U	0.18	U	0.26	UJ	0.26	U	0.25	U
Bromodichloromethane	UG/L	0	0%	MCL	80	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.17	U	0.17	U	0.33	UJ	0.33	U	0.25	U
Bromoform	UG/L	0	0%	MCL	80	0	0	107	1	U	2	U	2	U	1	UJ	1	UJ	1.1	U	0.2	U	0.2	U	0.23	UJ	0.23	U	0.5	UJ
Carbon disulfide	UG/L	0.61	2%				2	107	1	U	1	U	1	U	1	U	1	U	1	U	0.36	U	0.35	U	0.25	U	0.25	U	0.6	U
Carbon tetrachloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.36	U	0.36	U	0.22	UJ	0.22	U	0.5	UJ
Chlorobenzene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.26	U	0.26	U	0.22	UJ	0.22	U	0.25	U
Chlorodibromomethane	UG/L	0	0%	MCL	80	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.11	U	0.11	U	0.3	UJ	0.3	U	0.1	UJ
Chloroethane	UG/L	0.67	2%	GA	5	0	2	107	1	U	1	U	1	U	2	U	2	U	1	U	0.21	U	0.21	U	0.55	U	0.55	U	2	UJ
Chloroform	UG/L	0.32	1%	GA	7	0	1	107	1	U	1	U	1	U	1	U	1	U	1	U	0.16	U	0.16	U	0.32	UJ	0.32	U	0.14	U
Cis-1,2-Dichloroethene	UG/L	19	14%	GA	5	4	15	107	1	U	1	U	1	U	1	U	1	U	1	U	0.14	U	0.14	U	0.21	UJ	0.21	U	0.15	U
Cis-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.14	U	0.14	U	0.19	UJ	0.19	UJ	0.11	U
Cyclohexane	UG/L	8.6	11%				11	102	1	U					1	U	1	U	1	U	0.14	U	0.14	U	0.31	UJ	0.31	U	0.25	U
Dichlorodifluoromethane	UG/L	0	0%	GA	5	0	0	82	1	U					1	UJ	1	UJ	1	U	0.18	U	0.18	U					0.25	U
Diisoproply Ether	UG/L	0	0%				0	20																	0.21	U	0.21	U		
Ethyl benzene	UG/L	26	16%	GA	5	12	17	107	1	U	1	U	1	U	1	U	1	U	1	U	0.42	U	0.42	U	0.21	UJ	0.21	U	0.11	U
Isopropylbenzene	UG/L	2.6	9%	GA	5	0	10	107	1	U	1	U	1	U	1	U	1	U	1	U	0.34	U	0.34	U	0.23	UJ	0.23	U	0.1	U
Meta/Para Xylene	UG/L	19	11%	GA	5	4	7	61			2	U	2	U	1	U	1	U	2	U	0.81	U	0.81	U	0.59	UJ	0.59	U		
Methyl Acetate	UG/L	0	0%				0	102	1	U					10	U	10	U	2	U	0.48	U	0.47	U	0.53	U	0.53	U	0.19	UJ
Methyl bromide	UG/L	0	0%	GA	5	0	0	107	1	UJ	1	U	1	U	2	U	2	U	1	U	0.4	U	0.4	U	0.49	U	0.49	UJ	2	U
Methyl butyl ketone	UG/L	1.9	1%				1	107	5	U	2	U	2	U	5	U	5	U	5	U	0.4	U	0.4	U	1.7	U	1.7	U	1	U
Methyl chloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	2	U	2	U	1	U	0.18	U	0.18	U	0.36	U	0.36	UJ	0.33	U
Methyl cyclohexane	UG/L	4.2	7%				7	102	1	U					1	U	1	U	1	U	0.16	U	0.16	U	0.3	U	0.3	U	0.1	U
Methyl ethyl ketone	UG/L	9	10%				11	107	5	U	2	U	2	U	0.75	J	0.93	J	5	U	1	U	1	U	1.3	U	1.3	U	1	U
Methyl isobutyl ketone	UG/L	0	0%				0	107	5	U	1	U	1	U	5	U	5	U	5	U	0.34	U	0.34	U	1.3	U	1.3	U	1	U
Methyl Tertbutyl Ether	UG/L	0	0%				0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.13	U	0.13	U	0.36	U	0.36	U	0.2	U
Methylene chloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.13	U	0.13	U	1.1	U	1.1	U	1	U
Naphthalene	UG/L	0.23	4%				1	25			1	U	1	U											0.3	UJ	0.3	U		
n-Butylbenzene	UG/L	0	0%	GA	5	0	0	20																	0.23	UJ	0.23	U		
Ortho Xylene	UG/L	6.4	8%	GA	5	3	5	61			1	U	1	U	1	U	1	U	1	U	0.4	U	0.4	U	0.25	UJ	0.25	U		
p-Isopropyltoluene	UG/L	0	0%	GA	5	0	0	10	1	U																				
Propylbenzene	UG/L	0	0%	GA	5	0	0	10	1	U																				
sec-Butylbenzene	UG/L	0	0%	GA	5	0	0	20																	0.21	UJ	0.21	U		
Styrene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.36	U	0.35	U	0.23	UJ	0.23	U	0.11	U
tert-Butylbenzene	UG/L	0	0%	GA	5	0	0	20																	0.31	UJ	0.31	U		
Tetrachloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.42	U	0.42	U	0.4	U	0.4	U	0.15	U
Toluene	UG/L	14	11%	GA	5	5	12	107	1	U	1	U	1	U	1	U	1	U	1	U	0.21	U	0.21	U	0.27	UJ	0.27	U	0.33	U
Total Xylenes	UG/L	62	11%	GA	5	3	7	66	3	U															0.25	UJ	0.25	U	0.2	U
Trans-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.16	U	0.16	U	0.25	U	0.25	U	0.2	U
Trans-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	1	U	1	U	1	U	1	U	1	U	1	U	0.17	U	0.17	U	0.2	UJ	0.2	UJ	0.21	U
Trichloroethene	UG/L	2	10%	GA	5	0	11	107	1	U	1	U	1	U	1	U	1	U	1	U	0.19	U	0.19	U	0.28	UJ	0.28	U	0.13	U
Trichlorofluoromethane	UG/L	0	0%	GA	5	0	0	102	1	U					1	U	1	U	1	U	0.16	U	0.16	U	0.24	U	0.24	U	0.25	U
Vinyl chloride	UG/L	2.6	6%	GA	2	2	6	107	1	U	1	U	1	U	1	U	1	U	1	U	0.22	U	0.22	U	0.25	U	0.25	U	0.18	U

Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-25 MW25-17 GROUNDWATER 25LM20018 0-0.1 8/11/2006 SA LTM 2 Total Value	Qual	SEAD-25 MW25-17 GROUNDWATER 25LM20024 0-0.1 6/7/2007 DU LTM 3 Total Value	Qual	SEAD-25 MW25-17 GROUNDWATER 25LM20028 0-0.1 6/7/2007 SA LTM 3 Total Value	Qual	SEAD-25 MW25-17 GROUNDWATER 25LM20032 0-0.1 3/4/2008 DU LTM 4 Total Value	Qual	SEAD-25 MW25-17 GROUNDWATER 25LM20033 0-0.1 3/4/2008 SA LTM 4 Total Value	Qual	SEAD-25 MW25-17 GROUNDWATER 25LM20043 0-0.1 4/28/2009 SA LTM 5 Total Value	Qual	SEAD-25 MW25-17 GROUNDWATER 25LM20055 0-0.1 1/14/2010 SA LTM 6 Total Value	Qual	SEAD-25 MW25-17 GROUNDWATER 25LM20065 0-0 8/5/2010 SA LTM 7 Total Value	Qual	SEAD-25 MW25-17 GROUNDWATER 25LM20076 10.3-10.3 2/10/2011 SA LTM 8 Total Value	Qual	SEAD-25 MW25-17 GROUNDWATER 25LM20088 10.23-10.23 2/28/2012 SA LTM 9 Total Value	Qual	SEAD-25 MW25-17 GROUNDWATER 25LM20098 10.25-10.25 5/8/2013 SA LTM 10 Total Value	Qual	GR
Semivolatile Organic Compounds																																				
1,1'-Biphenyl	UG/L	0	0%	GA	5	0	0	18	10	U																										
2,4,5-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18	10	U																										
2,4,6-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18	10	U																										
2,4-Dichlorophenol	UG/L	0	0%	GA	5	0	0	18	10	U																										
2,4-Dimethylphenol	UG/L	0	0%				0	18	10	U																										
2,4-Dinitrophenol	UG/L	0	0%				0	18	49	U																										
2,4-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18	10	U																										
2,6-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18	10	U																										
2-Chloronaphthalene	UG/L	0	0%				0	18	10	U																										
2-Chlorophenol	UG/L	0	0%				0	18	10	U																										
2-Methylnaphthalene	UG/L	0	0%				0	18	10	U																										
2-Methylphenol	UG/L	0	0%				0	18	10	U																										
2-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	49	U																										
2-Nitrophenol	UG/L	0	0%	GA	1	0	0	18	10	U																										
3,3'-Dichlorobenzidine	UG/L	0	0%	GA	5	0	0	18	20	U																										
3-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	49	U																										
4,6-Dinitro-2-methylphenol	UG/L	0	0%	GA	1	0	0	18	49	U																										
4-Bromophenyl phenyl ether	UG/L	0	0%				0	18	10	U																										
4-Chloro-3-methylphenol	UG/L	0	0%	GA	1	0	0	18	10	U																										
4-Chloroaniline	UG/L	0	0%	GA	5	0	0	18	10	U																										
4-Chlorophenyl phenyl ether	UG/L	0	0%				0	18	10	U																										
4-Methylphenol	UG/L	0	0%				0	18	10	U																										
4-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	49	U																										
4-Nitrophenol	UG/L	0	0%	GA	1	0	0	18	49	U																										
Acenaphthene	UG/L	0.5	6%				1	18	10	U																										
Acenaphthylene	UG/L	2	22%				4	18	10	U																										
Acetophenone	UG/L	0	0%				0	18	10	U																										
Anthracene	UG/L	1	6%				1	18	10	U																										
Atrazine	UG/L	0	0%	GA	7.5	0	0	18	10	U																										
Benzaldehyde	UG/L	0	0%				0	18	49	U																										
Benzo(a)anthracene	UG/L	0	0%				0	18	10	U																										
Benzo(a)pyrene	UG/L	0	0%	GA	0		0	18	10	U																										
Benzo(b)fluoranthene	UG/L	0	0%				0	18	10	U																										
Benzo(ghi)perylene	UG/L	0.6	6%				1	18	10	U																										
Benzo(k)fluoranthene	UG/L	0	0%				0	18	10	U																										
Bis(2-Chloroethoxy)methane	UG/L	0	0%	GA	5	0	0	18	10	U																										
Bis(2-Chloroethyl)ether	UG/L	0	0%	GA	1	0	0	18	10	U																										
Bis(2-Chloroisopropyl)ether	UG/L	0	0%	GA	5	0	0	18	10	U																										
Bis(2-Ethylhexyl)phthalate	UG/L	11	6%	GA	5	1	1	18	10	U																										
Butylbenzylphthalate	UG/L	2	6%				1	18	10	U																										
Caprolactam	UG/L	0	0%				0	18	10	U																										
Carbazole	UG/L	0	0%				0	18	10	U																										
Chrysene	UG/L	0	0%				0	18	10	U																										
Dibenz(a,h)anthracene	UG/L	0	0%				0	18	10	U																										
Dibenzofuran	UG/L	0	0%				0	18	10	U																										
Diethyl phthalate	UG/L	0	0%				0	18	10	U																										
Dimethylphthalate	UG/L	0	0%				0	18	10	U																										
Di-n-butylphthalate	UG/L	0	0%	GA	50	0	0	18	10	U																										
Di-n-octylphthalate	UG/L	0	0%				0	18	10	U																										
Fluoranthene	UG/L	0	0%				0	18	10	U																										
Fluorene	UG/L	0	0%				0	18	10	U																										
Hexachlorobenzene	UG/L	0	0%	GA	0.04	0	0	18	10	U																										
Hexachlorobutadiene	UG/L	0	0%	GA	0.5	0	0	18	10	U																										
Hexachlorocyclopentadiene	UG/L	0	0%	GA	5	0	0	18	44	U																										
Hexachloroethane	UG/L	0	0%	GA	5	0	0	18	10	U																										
Indeno(1,2,3-cd)pyrene	UG/L	0	0%				0	18	10	U																										
Isophorone	UG/L	0	0%				0	18	10	U																										
Naphthalene	UG/L	2	6%				1	18	10	U																										
Nitrobenzene	UG/L	0	0%	GA	0.4	0	0	18	10	U																										
N-Nitroso-di-n-propylamine	UG/L	0	0%				0	18	10	U																										
N-Nitrosodiphenylamine	UG/L	0	0%				0	18	10	U																										
Pentachlorophenol	UG/L	0	0%	GA	1	0	0	18	49	U																										
Phenanthrene	UG/L	0	0%				0	18	10	U																										
Phenol	UG/L	0	0%	GA	1	0	0	18	10	U																										
Pyrene	UG/L	0	0%				0	18	10	U																										

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		
Loc ID									MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		MW25-17		
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GR
Sample ID									25LM20018		25LM20024		25LM20028		25LM20032		25LM20033		25LM20043		25LM20055		25LM20065		25LM20076		25LM20088		25LM20098	
Sample Depth Interval (FT)									0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0		10.3-10.3		10.23-10.23		10.25-10.25	
Sample Date									8/11/2006		6/7/2007		6/7/2007		3/4/2008		3/4/2008		4/28/2009		1/14/2010		8/5/2010		2/10/2011		2/28/2012		5/8/2013	
QC Type									SA		DU		SA		DU		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									2		3		3		4		4		5		6		7		8		9		10	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																														
Iron	UG/L	15,700	85%	GA	300	56	84	99	8.8	U	**390**	**J**	**490**	**J**	100	U	100	U	160		86.9	J	56.4	J	15.9	J	22.4	J	50	U
Sodium	UG/L	58,100	100%	GA	20,000	14	99	99	5,170	J	7,700	J	9,300	J	4,690		4,410		4,700		4,450		5,650		4,470		4,370		5,500	J
Wet Chemistry - MEE																														
Chloride	MG/L	97.9	78%	GA	250	0	78	100	1.4	J	3.5		3.7		0.2	U	0.2	U	0.2	U	2.5		5.3		2.3		0.47	J	1	U
Ethane	UG/L	1.1	5%				5	104	2	U	0.21		0.25		1	U	1	U	1	U	0.21	U	0.16	U	0.58	U	0.58	U	0.81	U
Ethene	UG/L	4.6	5%				5	104	2	U	1.2		1.4		1	U	1	U	1	U	0.22	U	0.17	U	0.69	U	0.69	U	0.73	U
Methane	UG/L	170	57%				59	104	2	U	6.1		7		2	U	2	U	2	U	0.14	U	0.14	U	0.98	J	0.93	J	0.45	U
Nitrate	MG/L	6.4	68%	GA	10	0	45	66			6.4	J	0.48	J					0.05	U	0.245	J			0.27		0.12		0.19	
Nitrate Nitrogen	MG/L	1	45%				13	29	0.11						0.798	J	1	J												
Nitrate/Nitrite Nitrogen	MG/L	1	64%	GA	10	0	16	25							0.798		1				0.245	J	0.484	J						
Nitrite	MG/L	0.73	27%	GA	1	0	18	66			0.73	J	0.5	UJ					0.01	U	0.007	UJ			0.00321	U	0.015	J	0.01	U
Nitrite Nitrogen	MG/L	0.087	3%				1	29	0.05	U					0.01	UJ	0.01	UJ												
Sulfate	MG/L	182	100%	GA	250	0	100	100	16.3		19		18		19.6		19.1		17.3		16.7	J	21.7		16	J	11	J	18	J
Field Measurement - Hach Kit																														
Conductivity	S/m	1.26	100%				90	90	0.593		0.418		0.418		0.532		0.532		0.379		0.418		0.584				0.423		0.558	
Conductivity (post)	S/m	0.844	100%				3	3																	0.599					
Conductivity (pre)	S/m	0.83	100%				3	3																	0.547					
Dissolved Oxygen	MG/L	12.6	100%				86	86	5.31		0.31		0.31		8.24		8.24		7.45		6.79		4.1				6.91		6.52	
Dissolved Oxygen (post)	MG/L	5.17	100%				3	3																	5.17					
Dissolved Oxygen (pre)	MG/L	5.36	100%				3	3																	5.36					
Nitrate Nitrogen	MG/L	0.5	100%				3	3																	0.1					
Nitrite Nitrogen	MG/L	0.5	100%				3	3																	0.004					
ORP	mV	259	100%				90	90	157		134		134		155		155		192		211		61				196		73	
ORP (post)	mV	197	100%				3	3																	192					
ORP (pre)	mV	193	100%				3	3																	193					
pH	Std units	8.37	100%				90	90	6.72		7.2		7.2		7.3		7.3		7.31		7.29		7.25				7.48		7.76	
pH (post)	Std units	7.38	100%				3	3																	7.38					
pH (pre)	Std units	7.38	100%				3	3																	7.38					
Sulfide	MG/L	1.04	89%				72	81	0.01	U	0.06		0.06		0.01		0.01		0.01	U	0		0		0		0		0.01	
Temperature	DEG C	26.55	100%				93	93	18.27		13.2		13.2		6		6		7.2		8.1		17.6		6.4		6.5		7.4	
Turbidity	NTU	195	100%				90	90	1.7		12		12		2.03		2.03		1.2		1.4		2.45				3.47		2.48	
Turbidity (post)	NTU	7.6	100%				2	2																	0					
Turbidity (pre)	NTU	5.7	100%				1	1																	0					

Notes:
1. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
2. Shading indicates concentration above cleanup goal.

U = compound was not detected
J = the reported value is an estimated concentration
UJ = the compound was not detected; the associated reporting limit is approximate
ND = Non-detect

SA = Sample
DU = Duplicate

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-17		MW25-17		MW25-17		MW25-18		MW25-18		MW25-18		MW25-18		MW25-18		MW25-18		MW25-18	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20108		25LM20111		25LM20112		25LM20118		25LM20009		25LM20019		25LM20029		25LM20034		25LM20044		25LM20056		25LM20066	
Sample Depth Interval (FT)									10.27-10.27		10.27-10.27		10.24-10.24		10.1-10.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0
Sample Date									6/18/2014		6/18/2014		3/17/2015		3/16/2016		1/30/2006		8/14/2006		6/6/2007		3/5/2008		4/28/2009		1/14/2010		8/5/2010	
QC Type									SA		DU		SA		SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									11		11		12		13		1		2		3		4		5		6		7	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Volatile Organic Compounds																														
1,1,1-Trichloroethane	UG/L	0.62	2%	GA	5	0	2	107	0.5	U	0.5	U	0.37	U	0.37	U	1	U	1	U	1	UJ	1	U	1	U	0.32	U	0.32	U
1,1,2,2-Tetrachloroethane	UG/L	0	0%	GA	5	0	0	107	0.18	U	0.18	U	0.62	U	0.62	U	1	U	1	U	1	UJ	1	U	1	U	0.09	U	0.09	U
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/L	0	0%	GA	5	0	0	102	0.5	U	0.5	U	0.36	U	0.36	U	1	UJ	1	U			1	U	1	U	0.4	U	0.4	U
1,1,2-Trichloroethane	UG/L	0	0%	GA	1	0	0	107	0.13	U	0.13	U	0.33	U	0.33	U	1	U	1	U	1	UJ	1	U	1	U	0.2	U	0.2	U
1,1-Dichloroethane	UG/L	3.5	9%	GA	5	0	10	107	0.25	U	0.25	U	0.38	U	0.38	U	1	U	1	U	1	UJ	1	U	1	U	0.14	U	0.14	U
1,1-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	0.11	U	0.11	U	0.36	U	0.36	U	1	U	1	U	1	UJ	1	U	1	U	0.37	U	0.37	U
1,2,4-Trichlorobenzene	UG/L	0	0%	GA	5	0	0	102	0.25	UJ	0.25	U	2.5	U	2.5	U	1	U	1	U			1	U	1	U	0.19	U	0.19	U
1,2,4-Trimethylbenzene	UG/L	0.45	7%	GA	5	0	2	30											1	U										
1,2-Dibromo-3-chloropropane	UG/L	0	0%	GA	0.04	0	0	102	0.44	U	0.44	U	1.1	U	1.1	U	1	U	1	U			2	U	2	U	0.43	U	0.43	U
1,2-Dibromoethane	UG/L	0	0%	GA	0.0006	0	0	107	0.25	U	0.25	U	0.44	U	0.44	U	1	U	1	U	1	UJ	1	U	1	U	0.18	U	0.18	U
1,2-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.21	U	0.21	U	0.37	U	0.37	U	1	U	1	U			1	U	1	U	0.4	U	0.4	U
1,2-Dichloroethane	UG/L	0.49	3%	GA	0.6	0	3	107	0.1	U	0.1	U	0.5	U	0.5	U	1	U	1	U	1	UJ	1	U	1	U	0.14	U	0.14	U
1,2-Dichloroethene (total)	UG/L	15	15%	GA	5	2	3	20																						
1,2-Dichloropropane	UG/L	0	0%	GA	1	0	0	107	0.13	U	0.13	U	0.67	U	0.67	U	1	U	1	U	1	UJ	1	U	1	U	0.15	U	0.15	U
1,3,5-Trimethylbenzene	UG/L	0	0%	GA	5	0	0	30											1	U										
1,3-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.25	U	0.25	U	0.43	U	0.43	U	1	U	1	U			1	U	1	U	0.36	U	0.36	U
1,4-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.28	U	0.28	U	0.46	U	0.46	U	1	U	1	U			1	U	1	U	0.34	U	0.34	U
Acetone	UG/L	11	7%				7	107	5	U	5	U	7	U	7	U	5	U	5	U	5	UJ	10	UJ	5	U	5	U	1.6	U
Benzene	UG/L	62	29%	GA	1	19	31	107	0.25	U	0.25	U	0.43	U	0.43	U	1	U	1	U	1	UJ	1	U	1	U	0.18	U	0.18	U
Bromodichloromethane	UG/L	0	0%	MCL	80	0	0	107	0.25	U	0.25	U	0.44	U	0.44	U	1	U	1	U	1	UJ	1	U	1	U	0.17	U	0.17	U
Bromoform	UG/L	0	0%	MCL	80	0	0	107	0.5	U	0.5	U	0.43	U	0.43	U	1	U	1	U	2	UJ	1	UJ	1.1	U	0.2	U	0.2	U
Carbon disulfide	UG/L	0.61	2%				2	107	0.6	U	0.6	U	1	U	1	U	1	U	1	U	1	UJ	1	U	1	U	0.36	U	0.35	U
Carbon tetrachloride	UG/L	0	0%	GA	5	0	0	107	0.5	U	0.5	U	0.33	U	0.33	U	1	U	1	U	1	UJ	1	U	1	U	0.36	U	0.36	U
Chlorobenzene	UG/L	0	0%	GA	5	0	0	107	0.25	U	0.25	U	0.26	U	0.26	U	1	U	1	U	1	UJ	1	U	1	U	0.26	U	0.26	U
Chlorodibromomethane	UG/L	0	0%	MCL	80	0	0	107	0.1	U	0.1	U	0.32	U	0.32	U	1	U	1	U	1	UJ	1	U	1	U	0.11	U	0.11	U
Chloroethane	UG/L	0.67	2%	GA	5	0	2	107	2	U	2	UJ	2.5	U	2.5	U	1	U	1	U	1	UJ	2	U	1	U	0.21	U	0.21	U
Chloroform	UG/L	0.32	1%	GA	7	0	1	107	0.14	U	0.14	U	0.5	U	0.5	U	1	U	1	U	1	UJ	1	U	1	U	0.16	U	0.16	U
Cis-1,2-Dichloroethene	UG/L	19	14%	GA	5	4	15	107	0.15	U	0.15	U	0.41	U	0.41	U	1	U	1	U	1	UJ	1	U	1	U	0.14	U	0.14	U
Cis-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	0.11	U	0.11	U	0.4	U	0.4	U	1	U	1	U	1	UJ	1	U	1	U	0.14	U	0.14	U
Cyclohexane	UG/L	8.6	11%				11	102	0.25	U	0.25	U	0.39	U	0.39	U	1	UJ	1	U			1	U	1	U	0.14	U	0.14	U
Dichlorodifluoromethane	UG/L	0	0%	GA	5	0	0	82	0.25	U	0.25	U	0.6	UJ	0.6	U	1	U	1	U			1	UJ	1	U	0.18	U	0.18	U
Diisoproply Ether	UG/L	0	0%				0	20																						
Ethyl benzene	UG/L	26	16%	GA	5	12	17	107	0.11	U	0.11	U	0.33	U	0.33	U	1	U	1	U	1	UJ	1	U	1	U	0.42	U	0.42	U
Isopropylbenzene	UG/L	2.6	9%	GA	5	0	10	107	0.1	U	0.1	U	0.35	U	0.35	U	1	U	1	U	1	UJ	1	U	1	U	0.34	U	0.34	U
Meta/Para Xylene	UG/L	19	11%	GA	5	4	7	61													2	UJ	1	U	2	U	0.81	U	0.81	U
Methyl Acetate	UG/L	0	0%				0	102	0.19	U	0.19	U	1.8	U	1.8	U	1	U	1	U			10	U	2	U	0.48	U	0.47	U
Methyl bromide	UG/L	0	0%	GA	5	0	0	107	2	U	2	U	2.5	UJ	2.5	UJ	1	U	1	U	1	UJ	2	U	1	U	0.4	U	0.4	U
Methyl butyl ketone	UG/L	1.9	1%				1	107	1	U	1	U	2	U	2	U	5	U	5	U	2	U	5	U	5	U	0.4	U	0.4	U
Methyl chloride	UG/L	0	0%	GA	5	0	0	107	0.33	U	0.33	U	0.4	U	0.4	U	1	U	1	U	1	UJ	2	U	1	U	0.18	U	0.18	U
Methyl cyclohexane	UG/L	4.2	7%				7	102	0.1	U	0.1	U	0.43	U	0.43	U	1	U	1	U			1	U	1	U	0.16	U	0.16	U
Methyl ethyl ketone	UG/L	9	10%				11	107	1	U	1	U	3.4	U	3.4	U	5	U	5	U	2	UJ	0.5	J	5	U	1	U	1	U
Methyl isobutyl ketone	UG/L	0	0%				0	107	1	U	1	U	2.1	U	2.1	U	5	U	5	U	1	UJ	5	U	5	U	0.34	U	0.34	U
Methyl Tertbutyl Ether	UG/L	0	0%				0	107	0.2	U	0.2	U	0.3	U	0.3	U	1	U	1	U	1	UJ	1	U	1	U	0.13	U	0.13	U
Methylene chloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	2.5	U	2.5	U	1	U	1	U	1	UJ	1	U	1	U	0.13	U	0.13	U
Naphthalene	UG/L	0.23	4%				1	25													1	UJ								
n-Butylbenzene	UG/L	0	0%	GA	5	0	0	20																						
Ortho Xylene	UG/L	6.4	8%	GA	5	3	5	61													1	UJ	1	U	1	U	0.4	U	0.4	U
p-Isopropyltoluene	UG/L	0	0%	GA	5	0	0	10											1	U										
Propylbenzene	UG/L	0	0%	GA	5	0	0	10											1	U										
sec-Butylbenzene	UG/L	0	0%	GA	5	0	0	20																						
Styrene	UG/L	0	0%	GA	5	0	0	107	0.11	U	0.11	U	0.27	U	0.27	U	1	U	1	U	1	UJ	1	U	1	U	0.36	U	0.35	U
tert-Butylbenzene	UG/L	0	0%	GA	5	0	0	20																						
Tetrachloroethene	UG/L	0	0%	GA	5	0	0	107	0.15	U	0.15	U	0.74	U	0.74	U	1	U	1	U	1	UJ	1	U	1	U	0.42	U	0.42	U
Toluene	UG/L	14	11%	GA	5	5	12	107	0.33	U	0.33	U	0.48	U	0.48	U	1	U	1	U	1	UJ	1	U	1	U	0.21	U	0.21	U
Total Xylenes	UG/L	62	11%	GA	5	3	7	66	0.2	U	0.2	U	0.23	U	0.23	U	3	U	3	U										
Trans-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	0.2	U	0.2	U	0.37	U	0.37	U	1	U	1	U	1	UJ	1	U	1	U	0.16	U	0.16	U
Trans-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	0.21	U	0.21	U	0.42	U	0.42	U	1	U	1	U	1	UJ	1	U	1	U	0.17	U	0.17	U
Trichloroethene	UG/L	2	10%	GA	5	0	11	107	0.13	U	0.13	U	0.48	U	0.48	U	1	U	1	U	1	UJ	1	U	1	U	0.19	U	0.19	U
Trichlorofluoromethane	UG/L	0	0%	GA	5	0	0	102	0.25	U	0.25	U	0.42	U	0.42	U	1	U	1	U			1	U	1	U	0.16	U	0.16	U
Vinyl chloride	UG/L	2.6	6%	GA	2	2	6	107	0.18	U	0.18	U	0.5	U	0.5	U	1	U	1	U	1	UJ	1	U	1	U	0.22	U	0.22	U

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-17		MW25-17		MW25-17		MW25-17		MW25-18		MW25-18		MW25-18		MW25-18		MW25-18		MW25-18	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20108		25LM20111		25LM20112		25LM20118		25LM20009		25LM20019		25LM20029		25LM20034		25LM20044		25LM20056		25LM20066	
Sample Depth Interval (FT)									10.27-10.27		10.27-10.27		10.24-10.24		10.1-10.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0	
Sample Date									6/18/2014		6/18/2014		3/17/2015		3/16/2016		1/30/2006		8/14/2006		6/6/2007		3/5/2008		4/28/2009		1/14/2010		8/5/2010	
QC Type									SA		DU		SA		SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									11		11		12		13		1		2		3		4		5		6		7	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Semivolatile Organic Compounds																														
1,1'-Biphenyl	UG/L	0	0%	GA	5	0	0	18									10	U	10	U										
2,4,5-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18									10	U	10	U										
2,4,6-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18									10	U	10	U										
2,4-Dichlorophenol	UG/L	0	0%	GA	5	0	0	18									10	U	10	U										
2,4-Dimethylphenol	UG/L	0	0%				0	18									10	U	10	U										
2,4-Dinitrophenol	UG/L	0	0%				0	18									48	U	48	UJ										
2,4-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18									10	U	10	U										
2,6-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18									10	U	10	U										
2-Chloronaphthalene	UG/L	0	0%				0	18									10	U	10	U										
2-Chlorophenol	UG/L	0	0%				0	18									10	U	10	U										
2-Methylnaphthalene	UG/L	0	0%				0	18									10	U	10	U										
2-Methylphenol	UG/L	0	0%				0	18									10	U	10	U										
2-Nitroaniline	UG/L	0	0%	GA	5	0	0	18									48	U	48	U										
2-Nitrophenol	UG/L	0	0%	GA	1	0	0	18									10	U	10	U										
3,3'-Dichlorobenzidine	UG/L	0	0%	GA	5	0	0	18									19	U	19	U										
3-Nitroaniline	UG/L	0	0%	GA	5	0	0	18									48	U	48	U										
4,6-Dinitro-2-methylphenol	UG/L	0	0%	GA	1	0	0	18									48	U	48	U										
4-Bromophenyl phenyl ether	UG/L	0	0%				0	18									10	U	10	U										
4-Chloro-3-methylphenol	UG/L	0	0%	GA	1	0	0	18									10	U	10	U										
4-Chloroaniline	UG/L	0	0%	GA	5	0	0	18									10	U	10	U										
4-Chlorophenyl phenyl ether	UG/L	0	0%				0	18									10	U	10	U										
4-Methylphenol	UG/L	0	0%				0	18									10	U	10	U										
4-Nitroaniline	UG/L	0	0%	GA	5	0	0	18									48	U	48	U										
4-Nitrophenol	UG/L	0	0%	GA	1	0	0	18									48	UJ	48	U										
Acenaphthene	UG/L	0.5	6%				1	18									10	U	10	U										
Acenaphthylene	UG/L	2	22%				4	18									10	U	10	U										
Acetophenone	UG/L	0	0%				0	18									10	U	10	U										
Anthracene	UG/L	1	6%				1	18									10	U	10	U										
Atrazine	UG/L	0	0%	GA	7.5	0	0	18									10	U	10	U										
Benzaldehyde	UG/L	0	0%				0	18									48	U	48	U										
Benzo(a)anthracene	UG/L	0	0%				0	18									10	U	10	U										
Benzo(a)pyrene	UG/L	0	0%	GA	0		0	18									10	U	10	U										
Benzo(b)fluoranthene	UG/L	0	0%				0	18									10	U	10	U										
Benzo(ghi)perylene	UG/L	0.6	6%				1	18									10	U	10	U										
Benzo(k)fluoranthene	UG/L	0	0%				0	18									10	U	10	U										
Bis(2-Chloroethoxy)methane	UG/L	0	0%	GA	5	0	0	18									10	U	10	U										
Bis(2-Chloroethyl)ether	UG/L	0	0%	GA	1	0	0	18									10	U	10	U										
Bis(2-Chloroisopropyl)ether	UG/L	0	0%	GA	5	0	0	18									10	U	10	U										
Bis(2-Ethylhexyl)phthalate	UG/L	11	6%	GA	5	1	1	18									10	U	**11**											
Butylbenzylphthalate	UG/L	2	6%				1	18									10	U	2	J										
Caprolactam	UG/L	0	0%				0	18									10	U	10	U										
Carbazole	UG/L	0	0%				0	18									10	U	10	U										
Chrysene	UG/L	0	0%				0	18									10	U	10	U										
Dibenz(a,h)anthracene	UG/L	0	0%				0	18									10	U	10	U										
Dibenzofuran	UG/L	0	0%				0	18									10	U	10	U										
Diethyl phthalate	UG/L	0	0%				0	18									10	U	10	U										
Dimethylphthalate	UG/L	0	0%				0	18									10	U	10	U										
Di-n-butylphthalate	UG/L	0	0%	GA	50	0	0	18									10	U	10	U										
Di-n-octylphthalate	UG/L	0	0%				0	18									10	U	10	UJ										
Fluoranthene	UG/L	0	0%				0	18									10	U	10	U										
Fluorene	UG/L	0	0%				0	18									10	U	10	U										
Hexachlorobenzene	UG/L	0	0%	GA	0.04	0	0	18									10	U	10	U										
Hexachlorobutadiene	UG/L	0	0%	GA	0.5	0	0	18									10	U	10	U										
Hexachlorocyclopentadiene	UG/L	0	0%	GA	5	0	0	18									43	U	44	U										
Hexachloroethane	UG/L	0	0%	GA	5	0	0	18									10	U	10	U										
Indeno(1,2,3-cd)pyrene	UG/L	0	0%				0	18									10	U	10	U										
Isophorone	UG/L	0	0%				0	18									10	U	10	U										
Naphthalene	UG/L	2	6%				1	18									10	U	10	U										
Nitrobenzene	UG/L	0	0%	GA	0.4	0	0	18									10	U	10	U										
N-Nitroso-di-n-propylamine	UG/L	0	0%				0	18									10	U	10	U										
N-Nitrosodiphenylamine	UG/L	0	0%				0	18									10	U	10	U										
Pentachlorophenol	UG/L	0	0%	GA	1	0	0	18									48	U	48	U										
Phenanthrene	UG/L	0	0%				0	18									10	U	10	U										
Phenol	UG/L	0	0%	GA	1	0	0	18									10	U	10	U										
Pyrene	UG/L	0	0%				0	18									10	U	10	U										

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-17		MW25-17		MW25-17		MW25-18		MW25-18		MW25-18		MW25-18		MW25-18		MW25-18	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20108		25LM20111		25LM20112		25LM20118		25LM20009		25LM20019		25LM20029		25LM20034		25LM20044		25LM20056		25LM20066	
Sample Depth Interval (FT)									10.27-10.27		10.27-10.27		10.24-10.24		10.1-10.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0	
Sample Date									6/18/2014		6/18/2014		3/17/2015		3/16/2016		1/30/2006		8/14/2006		6/6/2007		3/5/2008		4/28/2009		1/14/2010		8/5/2010	
QC Type									SA		DU		SA		SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									11		11		12		13		1		2		3		4		5		6		7	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																														
Iron	UG/L	15,700	85%	GA	300	56	84	99	50	U	50	U	50	U	17	U	**462**	**J**	**357**		**500**	**J**	107		100	J	122		83.8	J
Sodium	UG/L	58,100	100%	GA	20,000	14	99	99	7,500	J	4,900	J	5,200		3,500		**22,300**		**41,900**	**J**	**37,000**	**J**	**20,400**		19,000		**28,400**		**58,100**	
Wet Chemistry - MEE																														
Chloride	MG/L	97.9	78%	GA	250	0	78	100	0.59		0.59		1.2		0.33	J	18.6		55.6		59		18		16.3		51.7		97.9	
Ethane	UG/L	1.1	5%				5	104	0.55	U	0.55	U	0.55	U	0.55	U	2	U	2	U	0.024	J	1	U	1	U	0.16	U	0.16	U
Ethene	UG/L	4.6	5%				5	104	0.5	U	0.5	U	0.5	U	0.5	U	2	U	2	U	2		1	U	1	U	0.17	U	0.17	U
Methane	UG/L	170	57%				59	104	0.35	J	0.29	U	0.96		0.29	J	2	U	2	U	2		2	U	2	U	0.14	U	0.14	U
Nitrate	MG/L	6.4	68%	GA	10	0	45	66	0.17		0.17		0.24		0.18						1.5	J			0.05	U	0.2	J		
Nitrate Nitrogen	MG/L	1	45%				13	29									0.05	U	0.32				0.199	J						
Nitrate/Nitrite Nitrogen	MG/L	1	64%	GA	10	0	16	25															0.199				0.2	J	0.18	J
Nitrite	MG/L	0.73	27%	GA	1	0	18	66	0.01	U	0.01	U	0.01	U	0.01	U					0.5				0.01	U	0.007	UJ		
Nitrite Nitrogen	MG/L	0.087	3%				1	29									0.05	U	0.05	U			0.01	UJ						
Sulfate	MG/L	182	100%	GA	250	0	100	100	14		14		16		7.2		24.8		30.1		31		16.8		22.8		26.8	J	40.2	
Field Measurement - Hach Kit																														
Conductivity	S/m	1.26	100%				90	90					0.52		0.482		0.494		0.858		0.54		0.713		0.385		0.544		0.893	
Conductivity (post)	S/m	0.844	100%				3	3																						
Conductivity (pre)	S/m	0.83	100%				3	3																						
Dissolved Oxygen	MG/L	12.6	100%				86	86					5.59		6.24		3.99		6.21		0.96		4.68		4.43		4.39		2.1	
Dissolved Oxygen (post)	MG/L	5.17	100%				3	3																						
Dissolved Oxygen (pre)	MG/L	5.36	100%				3	3																						
Nitrate Nitrogen	MG/L	0.5	100%				3	3																						
Nitrite Nitrogen	MG/L	0.5	100%				3	3																						
ORP	mV	259	100%				90	90					224		170		63		46		98		144		150		237		123	
ORP (post)	mV	197	100%				3	3																						
ORP (pre)	mV	193	100%				3	3																						
pH	Std units	8.37	100%				90	90					7.51		8.37		7.62		7.32		7.15		7.31		7.3		7.28		7.21	
pH (post)	Std units	7.38	100%				3	3																						
pH (pre)	Std units	7.38	100%				3	3																						
Sulfide	MG/L	1.04	89%				72	81	0.01		0.01		0.12		0.02		1.04		0.01		0.01	U	0.06		0.01					
Temperature	DEG C	26.55	100%				93	93	5		5.6		7.2		24.41		13		4.9		7.1		8		19.3					
Turbidity	NTU	195	100%				90	90	1.65		0.75		31.8		6.22		11		5.04		11		2.78		3.12					
Turbidity (post)	NTU	7.6	100%				2	2																						
Turbidity (pre)	NTU	5.7	100%				1	1																						

Notes:
1. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
2. Shading indicates concentration above cleanup goal.

U = compound was not detected
J = the reported value is an estimated concentration
UJ = the compound was not detected; the associated reporting limit is approximate
ND = Non-detect

SA = Sample
DU = Duplicate

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-18		MW25-18		MW25-19		MW25-19		MW25-19		MW25-19		MW25-19		MW25-19		MW25-19		MW25-19	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20077		25LM20089		25LM20099		25LM20030		25LM20035		25LM20045		25LM20057		25LM20067		25LM20078		25LM20090		25LM20100	
Sample Depth Interval (FT)									10.18-10.18		10.15-10.15		10.2-10.2		0-0.1		0-0.1		0-0.1		0-0.1		0-0		11-11		10.98-10.98		11-11	
Sample Date									2/10/2011		2/29/2012		5/8/2013		6/7/2007		3/3/2008		4/28/2009		1/13/2010		8/4/2010		2/9/2011		2/28/2012		5/7/2013	
QC Type									SA		SA		SA		SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									8		9		10		3		4		5		6		7		8		9		10	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Volatile Organic Compounds																														
1,1,1-Trichloroethane	UG/L	0.62	2%	GA	5	0	2	107	0.2	UJ	0.2	U	0.5	U	1	U	1	U	1	U	0.32	U	0.8	U	0.2	UJ	0.2	U	0.5	UJ
1,1,2,2-Tetrachloroethane	UG/L	0	0%	GA	5	0	0	107	0.38	UJ	0.38	U	0.18	U	1	U	1	U	1	U	0.09	U	0.23	U	0.38	UJ	0.38	U	0.18	U
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/L	0	0%	GA	5	0	0	102	0.31	U	0.31	UJ	0.5	U			1	U	1	U	0.4	U	1	U	0.31	U	0.31	UJ	0.5	U
1,1,2-Trichloroethane	UG/L	0	0%	GA	1	0	0	107	0.33	UJ	0.33	U	0.13	U	1	U	1	U	1	U	0.2	U	0.5	U	0.33	UJ	0.33	U	0.13	U
1,1-Dichloroethane	UG/L	3.5	9%	GA	5	0	10	107	0.21	UJ	0.21	U	0.25	U	1	U	1	U	1	U	0.14	U	0.36	U	0.21	UJ	0.21	U	0.25	U
1,1-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	0.35	UJ	0.35	U	0.11	U	1	U	1	U	1	U	0.37	U	0.93	U	0.35	UJ	0.35	U	0.11	U
1,2,4-Trichlorobenzene	UG/L	0	0%	GA	5	0	0	102	0.37	UJ	0.37	U	0.25	UJ			1	U	1	U	0.19	U	0.48	U	0.37	UJ	0.37	U	0.25	U
1,2,4-Trimethylbenzene	UG/L	0.45	7%	GA	5	0	2	30	0.19	UJ	0.19	U													0.19	UJ	0.19	U		
1,2-Dibromo-3-chloropropane	UG/L	0	0%	GA	0.04	0	0	102	0.5	U	0.5	U	0.44	U			2	U	2	U	0.43	U	1.1	U	0.5	U	0.5	U	0.44	U
1,2-Dibromoethane	UG/L	0	0%	GA	0.0006	0	0	107	0.22	UJ	0.22	U	0.25	U	1	U	1	U	1	U	0.18	U	0.45	U	0.22	UJ	0.22	U	0.25	U
1,2-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.15	UJ	0.15	U	0.21	U			1	U	1	U	0.4	U	1	U	0.15	UJ	0.15	U	0.21	U
1,2-Dichloroethane	UG/L	0.49	3%	GA	0.6	0	3	107	0.2	UJ	0.2	U	0.1	U	1	U	1	U	1	U	0.14	U	0.36	U	0.2	UJ	0.2	U	0.1	U
1,2-Dichloroethene (total)	UG/L	15	15%	GA	5	2	3	20	0.21	UJ	0.21	UJ													0.21	UJ	0.21	UJ		
1,2-Dichloropropane	UG/L	0	0%	GA	1	0	0	107	0.25	UJ	0.25	U	0.13	U	1	U	1	U	1	U	0.15	U	0.38	U	0.25	UJ	0.25	U	0.13	U
1,3,5-Trimethylbenzene	UG/L	0	0%	GA	5	0	0	30	0.2	UJ	0.2	U													0.2	UJ	0.2	U		
1,3-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.26	UJ	0.26	U	0.25	U			1	U	1	U	0.36	U	0.9	U	0.26	UJ	0.26	U	0.25	U
1,4-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.24	UJ	0.24	U	0.28	U			1	U	1	U	0.34	U	0.86	U	0.24	UJ	0.24	U	0.28	U
Acetone	UG/L	11	7%				7	107	2.2	U	2.2	U	5	U	5	U	10	UJ	1.4	J	5	U	4	U	2.2	U	2.2	U	5	U
Benzene	UG/L	62	29%	GA	1	19	31	107	0.26	UJ	0.26	U	0.25	U	1	U	1	U	1	U	0.18	U	0.45	U	0.26	UJ	0.26	U	0.25	U
Bromodichloromethane	UG/L	0	0%	MCL	80	0	0	107	0.33	UJ	0.33	U	0.25	U	1	U	1	U	1	U	0.17	U	0.43	U	0.33	UJ	0.33	U	0.25	UJ
Bromoform	UG/L	0	0%	MCL	80	0	0	107	0.23	UJ	0.23	U	0.5	UJ	2	U	1	UJ	1	U	0.2	U	0.5	U	0.23	UJ	0.23	U	0.5	U
Carbon disulfide	UG/L	0.61	2%				2	107	0.25	U	0.25	U	0.6	U	1	U	1	U	1	U	0.36	U	0.88	U	0.25	U	0.25	U	0.6	U
Carbon tetrachloride	UG/L	0	0%	GA	5	0	0	107	0.22	UJ	0.22	U	0.5	UJ	1	U	1	U	1	U	0.36	U	0.9	U	0.22	UJ	0.22	U	0.5	UJ
Chlorobenzene	UG/L	0	0%	GA	5	0	0	107	0.22	UJ	0.22	U	0.25	U	1	U	1	U	1	U	0.26	U	0.65	U	0.22	UJ	0.22	U	0.25	U
Chlorodibromomethane	UG/L	0	0%	MCL	80	0	0	107	0.3	UJ	0.3	U	0.1	UJ	1	U	1	U	1	U	0.11	U	0.28	U	0.3	UJ	0.3	U	0.1	U
Chloroethane	UG/L	0.67	2%	GA	5	0	2	107	0.55	U	0.55	U	2	UJ	1	U	2	U	1	U	0.21	U	0.53	U	0.55	U	0.55	U	2	U
Chloroform	UG/L	0.32	1%	GA	7	0	1	107	0.32	UJ	0.32	U	0.14	U	1	U	1	U	1	U	0.16	U	0.4	U	0.32	UJ	0.32	U	0.14	U
Cis-1,2-Dichloroethene	UG/L	19	14%	GA	5	4	15	107	0.21	UJ	0.21	U	0.15	U	0.2	J	1	U	1	U	0.14	U	0.36	U	0.21	UJ	0.21	U	0.15	U
Cis-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	0.19	UJ	0.19	UJ	0.11	U	1	U	1	U	1	U	0.14	U	0.36	U	0.19	UJ	0.19	UJ	0.11	U
Cyclohexane	UG/L	8.6	11%				11	102	0.31	UJ	0.31	U	0.25	U			1	U	1	U	0.14	U	0.36	U	0.31	UJ	0.31	U	0.25	U
Dichlorodifluoromethane	UG/L	0	0%	GA	5	0	0	82					0.25	U			1	UJ	1	U	0.18	U	0.45	U					0.25	U
Diisoproply Ether	UG/L	0	0%				0	20	0.21	U	0.21	U													0.21	U	0.21	U		
Ethyl benzene	UG/L	26	16%	GA	5	12	17	107	0.21	UJ	0.21	U	0.11	U	1	U	1	U	1	U	0.42	U	1.1	U	0.21	UJ	0.21	U	0.11	U
Isopropylbenzene	UG/L	2.6	9%	GA	5	0	10	107	0.23	UJ	0.23	U	0.1	U	1	U	1	U	1	U	0.34	U	0.86	U	0.23	UJ	0.23	U	0.1	U
Meta/Para Xylene	UG/L	19	11%	GA	5	4	7	61	0.59	UJ	0.59	U			2	U	1	U	2	U	0.81	U	2.1	U	0.59	UJ	0.59	U		
Methyl Acetate	UG/L	0	0%				0	102	0.53	U	0.53	U	0.19	UJ			10	U	2	U	0.48	U	1.2	U	0.53	U	0.53	U	0.19	U
Methyl bromide	UG/L	0	0%	GA	5	0	0	107	0.49	U	0.49	UJ	2	U	1	U	2	U	1	U	0.4	U	1	U	0.49	U	0.49	UJ	2	U
Methyl butyl ketone	UG/L	1.9	1%				1	107	1.7	U	1.7	U	1	U	2	U	5	U	5	U	0.4	U	1	U	1.7	U	1.7	U	1	U
Methyl chloride	UG/L	0	0%	GA	5	0	0	107	0.36	U	0.36	U	0.33	U	1	U	2	U	1	U	0.18	U	0.45	U	0.36	U	0.36	UJ	0.33	U
Methyl cyclohexane	UG/L	4.2	7%				7	102	0.3	U	0.3	U	0.1	U			1	U	1	U	0.16	U	0.4	U	0.3	U	0.3	U	0.1	U
Methyl ethyl ketone	UG/L	9	10%				11	107	1.3	U	1.3	U	1	U	2	U	5	U	5	U	1	U	2.5	U	1.3	U	1.3	U	1	U
Methyl isobutyl ketone	UG/L	0	0%				0	107	1.3	U	1.3	U	1	U	1	U	5	U	5	U	0.34	U	0.86	U	1.3	U	1.3	U	1	U
Methyl Tertbutyl Ether	UG/L	0	0%				0	107	0.36	U	0.36	U	0.2	U	1	U	1	U	1	U	0.13	U	0.33	U	0.36	U	0.36	U	0.2	U
Methylene chloride	UG/L	0	0%	GA	5	0	0	107	1.1	U	1.1	U	1	U	1	U	1	U	1	U	0.13	U	0.33	U	1.1	U	1.1	U	1	U
Naphthalene	UG/L	0.23	4%				1	25	0.3	UJ	0.3	U			1	U									0.3	UJ	0.3	U		
n-Butylbenzene	UG/L	0	0%	GA	5	0	0	20	0.23	UJ	0.23	U													0.23	UJ	0.23	U		
Ortho Xylene	UG/L	6.4	8%	GA	5	3	5	61	0.25	UJ	0.25	U			1	U	1	U	1	U	0.4	U	1	U	0.25	UJ	0.25	U		
p-Isopropyltoluene	UG/L	0	0%	GA	5	0	0	10																						
Propylbenzene	UG/L	0	0%	GA	5	0	0	10																						
sec-Butylbenzene	UG/L	0	0%	GA	5	0	0	20	0.21	UJ	0.21	U													0.21	UJ	0.21	U		
Styrene	UG/L	0	0%	GA	5	0	0	107	0.23	UJ	0.23	U	0.11	U	1	U	1	U	1	U	0.36	U	0.88	U	0.23	UJ	0.23	U	0.11	U
tert-Butylbenzene	UG/L	0	0%	GA	5	0	0	20	0.31	UJ	0.31	U													0.31	UJ	0.31	U		
Tetrachloroethene	UG/L	0	0%	GA	5	0	0	107	0.4	U	0.4	U	0.15	U	1	U	1	U	1	U	0.42	U	1.1	U	0.4	U	0.4	U	0.15	U
Toluene	UG/L	14	11%	GA	5	5	12	107	0.27	UJ	0.27	U	0.33	U	1	U	1	U	1	U	0.21	U	0.53	U	0.27	UJ	0.27	U	0.33	U
Total Xylenes	UG/L	62	11%	GA	5	3	7	66	0.25	UJ	0.25	U	0.2	U											0.25	UJ	0.25	U	0.2	U
Trans-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	0.25	U	0.25	U	0.2	U	1	U	1	U	1	U	0.16	U	0.4	U	0.25	U	0.25	U	0.2	U
Trans-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	0.2	UJ	0.2	UJ	0.21	U	1	U	1	U	1	U	0.17	U	0.43	U	0.2	UJ	0.2	UJ	0.21	UJ
Trichloroethene	UG/L	2	10%	GA	5	0	11	107	0.28	UJ	0.28	U	0.13	U	1	U	1	U	1	U	0.19	U	0.48	U	0.28	UJ	0.28	U	0.13	U
Trichlorofluoromethane	UG/L	0	0%	GA	5	0	0	102	0.24	U	0.24	U	0.25	U			1	U	1	U	0.16	U	0.4	U	0.24	U	0.24	U	0.25	U
Vinyl chloride	UG/L	2.6	6%	GA	2	2	6	107	0.25	U	0.25	U	0.18	U	1	U	1	U	1	U	0.22	U	0.55	U	0.25	U	0.25	U	0.18	U

Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Area	Loc ID	Matrix	Sample ID	Sample Depth Interval (FT)	Sample Date	QC Type	Study ID	Sample Round	Filtered	Value	Qual
									SEAD-25	MW25-18	GROUNDWATER	25LM20077	10.18-10.18	2/10/2011	SA	LTM	8	Total		
									SEAD-25	MW25-18	GROUNDWATER	25LM20089	10.15-10.15	2/29/2012	SA	LTM	9	Total		
									SEAD-25	MW25-19	GROUNDWATER	25LM20099	10.2-10.2	5/8/2013	SA	LTM	10	Total		
									SEAD-25	MW25-19	GROUNDWATER	25LM20030	0-0.1	6/7/2007	SA	LTM	3	Total		
									SEAD-25	MW25-19	GROUNDWATER	25LM20035	0-0.1	3/3/2008	SA	LTM	4	Total		
									SEAD-25	MW25-19	GROUNDWATER	25LM20045	0-0.1	4/28/2009	SA	LTM	5	Total		
									SEAD-25	MW25-19	GROUNDWATER	25LM20057	0-0.1	1/13/2010	SA	LTM	6	Total		
									SEAD-25	MW25-19	GROUNDWATER	25LM20067	0-0	8/4/2010	SA	LTM	7	Total		
									SEAD-25	MW25-19	GROUNDWATER	25LM20078	11-11	2/9/2011	SA	LTM	8	Total		
									SEAD-25	MW25-19	GROUNDWATER	25LM20090	10.98-10.98	2/28/2012	SA	LTM	9	Total		
									SEAD-25	MW25-19	GROUNDWATER	25LM20100	11-11	5/7/2013	SA	LTM	10	Total		
Semivolatile Organic Compounds																				
1,1'-Biphenyl	UG/L	0	0%	GA	5	0	0	18												
2,4,5-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18												
2,4,6-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18												
2,4-Dichlorophenol	UG/L	0	0%	GA	5	0	0	18												
2,4-Dimethylphenol	UG/L	0	0%				0	18												
2,4-Dinitrophenol	UG/L	0	0%				0	18												
2,4-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18												
2,6-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18												
2-Chloronaphthalene	UG/L	0	0%				0	18												
2-Chlorophenol	UG/L	0	0%				0	18												
2-Methylnaphthalene	UG/L	0	0%				0	18												
2-Methylphenol	UG/L	0	0%				0	18												
2-Nitroaniline	UG/L	0	0%	GA	5	0	0	18												
2-Nitrophenol	UG/L	0	0%	GA	1	0	0	18												
3,3'-Dichlorobenzidine	UG/L	0	0%	GA	5	0	0	18												
3-Nitroaniline	UG/L	0	0%	GA	5	0	0	18												
4,6-Dinitro-2-methylphenol	UG/L	0	0%	GA	1	0	0	18												
4-Bromophenyl phenyl ether	UG/L	0	0%				0	18												
4-Chloro-3-methylphenol	UG/L	0	0%	GA	1	0	0	18												
4-Chloroaniline	UG/L	0	0%	GA	5	0	0	18												
4-Chlorophenyl phenyl ether	UG/L	0	0%				0	18												
4-Methylphenol	UG/L	0	0%				0	18												
4-Nitroaniline	UG/L	0	0%	GA	5	0	0	18												
4-Nitrophenol	UG/L	0	0%	GA	1	0	0	18												
Acenaphthene	UG/L	0.5	6%				1	18												
Acenaphthylene	UG/L	2	22%				4	18												
Acetophenone	UG/L	0	0%				0	18												
Anthracene	UG/L	1	6%				1	18												
Atrazine	UG/L	0	0%	GA	7.5	0	0	18												
Benzaldehyde	UG/L	0	0%				0	18												
Benzo(a)anthracene	UG/L	0	0%				0	18												
Benzo(a)pyrene	UG/L	0	0%	GA	0		0	18												
Benzo(b)fluoranthene	UG/L	0	0%				0	18												
Benzo(ghi)perylene	UG/L	0.6	6%				1	18												
Benzo(k)fluoranthene	UG/L	0	0%				0	18												
Bis(2-Chloroethoxy)methane	UG/L	0	0%	GA	5	0	0	18												
Bis(2-Chloroethyl)ether	UG/L	0	0%	GA	1	0	0	18												
Bis(2-Chloroisopropyl)ether	UG/L	0	0%	GA	5	0	0	18												
Bis(2-Ethylhexyl)phthalate	UG/L	11	6%	GA	5	1	1	18												
Butylbenzylphthalate	UG/L	2	6%				1	18												
Caprolactam	UG/L	0	0%				0	18												
Carbazole	UG/L	0	0%				0	18												
Chrysene	UG/L	0	0%				0	18												
Dibenz(a,h)anthracene	UG/L	0	0%				0	18												
Dibenzofuran	UG/L	0	0%				0	18												
Diethyl phthalate	UG/L	0	0%				0	18												
Dimethylphthalate	UG/L	0	0%				0	18												
Di-n-butylphthalate	UG/L	0	0%	GA	50	0	0	18												
Di-n-octylphthalate	UG/L	0	0%				0	18												
Fluoranthene	UG/L	0	0%				0	18												
Fluorene	UG/L	0	0%				0	18												
Hexachlorobenzene	UG/L	0	0%	GA	0.04	0	0	18												
Hexachlorobutadiene	UG/L	0	0%	GA	0.5	0	0	18												
Hexachlorocyclopentadiene	UG/L	0	0%	GA	5	0	0	18												
Hexachloroethane	UG/L	0	0%	GA	5	0	0	18												
Indeno(1,2,3-cd)pyrene	UG/L	0	0%				0	18												
Isophorone	UG/L	0	0%				0	18												
Naphthalene	UG/L	2	6%				1	18												
Nitrobenzene	UG/L	0	0%	GA	0.4	0	0	18												
N-Nitroso-di-n-propylamine	UG/L	0	0%				0	18												
N-Nitrosodiphenylamine	UG/L	0	0%				0	18												
Pentachlorophenol	UG/L	0	0%	GA	1	0	0	18												
Phenanthrene	UG/L	0	0%				0	18												
Phenol	UG/L	0	0%	GA	1	0	0	18												
Pyrene	UG/L	0	0%				0	18												

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-18		MW25-18		MW25-19		MW25-19		MW25-19		MW25-19		MW25-19		MW25-19		MW25-19		MW25-19	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20077		25LM20089		25LM20099		25LM20030		25LM20035		25LM20045		25LM20057		25LM20067		25LM20078		25LM20090		25LM20100	
Sample Depth Interval (FT)									10.18-10.18		10.15-10.15		10.2-10.2		0-0.1		0-0.1		0-0.1		0-0.1		0-0		11-11		10.98-10.98		11-11	
Sample Date									2/10/2011		2/29/2012		5/8/2013		6/7/2007		3/3/2008		4/28/2009		1/13/2010		8/4/2010		2/9/2011		2/28/2012		5/7/2013	
QC Type									SA		SA		SA		SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									8		9		10		3		4		5		6		7		8		9		10	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																														
Iron	UG/L	15,700	85%	GA	300	56	84	99	250		**446**	**J**	**440**	**J**	**1,200**	**J**	**515**		20	J	204		**1,310**		152		53.8	J	50	U
Sodium	UG/L	58,100	100%	GA	20,000	14	99	99	**41,900**		**27,300**		**22,000**	**J**	3,800	J	4,520		3,500		4,350		4,140		3,340		3,980		3,600	
Wet Chemistry - MEE																														
Chloride	MG/L	97.9	78%	GA	250	0	78	100	72		20	J	16		4.5		0.2	U	0.2	U	2.3		3.6		1.7	J	0.72	J	1	U
Ethane	UG/L	1.1	5%				5	104	0.58	U	0.58	U	4	U	1.1		1	U	1	U	0.16	U	0.16	U	0.58	U	0.58	U	0.81	U
Ethene	UG/L	4.6	5%				5	104	0.69	U	0.69	U	3	U	4.6		1	U	1	U	0.17	U	0.17	U	0.69	U	0.69	U	0.73	U
Methane	UG/L	170	57%				59	104	1.3	J	1.9	J	2	U	29		2	U	2	U	0.14	U	0.14	U	1.2	J	0.92	J	0.45	U
Nitrate	MG/L	6.4	68%	GA	10	0	45	66	0.18		0.13		0.18		1.4	J			0.05	U	0.113	J			0.064		0.094		0.13	
Nitrate Nitrogen	MG/L	1	45%				13	29									0.194	J												
Nitrate/Nitrite Nitrogen	MG/L	1	64%	GA	10	0	16	25									0.194				0.113	J	0.072	J						
Nitrite	MG/L	0.73	27%	GA	1	0	18	66	0.00321	U	0.022	J	0.01	U	0.72	J			0.01	U	0.007	UJ			0.00321	U	0.015	J	0.01	U
Nitrite Nitrogen	MG/L	0.087	3%				1	29									0.01	UJ												
Sulfate	MG/L	182	100%	GA	250	0	100	100	32	J	21	J	27	J	23		24.3		30.1		31	J	36.7		31	J	22	J	29	
Field Measurement - Hach Kit																														
Conductivity	S/m	1.26	100%				90	90			0.548		0.566		0.427		0.478		0.379		0.445		0.662				0.477		0.423	
Conductivity (post)	S/m	0.844	100%				3	3	0.844																0.592					
Conductivity (pre)	S/m	0.83	100%				3	3	0.83																0.588					
Dissolved Oxygen	MG/L	12.6	100%				86	86			3.89		5.26		0.05		5.84		3.75		4.01		0.03				2.3		2.23	
Dissolved Oxygen (post)	MG/L	5.17	100%				3	3	2.99																2.25					
Dissolved Oxygen (pre)	MG/L	5.36	100%				3	3	3.52																2.84					
Nitrate Nitrogen	MG/L	0.5	100%				3	3	0.5																0.008					
Nitrite Nitrogen	MG/L	0.5	100%				3	3	0.2																0.5					
ORP	mV	259	100%				90	90			70		48		117		161		134		259		63				70		178	
ORP (post)	mV	197	100%				3	3	185																197					
ORP (pre)	mV	193	100%				3	3	187																174					
pH	Std units	8.37	100%				90	90			7.16		7.81		7.04		7.23		7.15		7.08		6.94				6.95		7.48	
pH (post)	Std units	7.38	100%				3	3	7.29																7.22					
pH (pre)	Std units	7.38	100%				3	3	7.3																7.22					
Sulfide	MG/L	1.04	89%				72	81	0.03		0.01		0.05		0.1		0.01		0.02		0.02		0.18		0.03		0.016		0	
Temperature	DEG C	26.55	100%				93	93	6.3		6.7		7.6		13.4		5.8		7.1		8		17.7		6.9		6.6		7.5	
Turbidity	NTU	195	100%				90	90			3.66		7.55		17		16.4		1.3		6.1		1.4				2.35		0.43	
Turbidity (post)	NTU	7.6	100%				2	2	7.6																1.2					
Turbidity (pre)	NTU	5.7	100%				1	1	0																5.7					

Notes:
1. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
2. Shading indicates concentration above cleanup goal.

U = compound was not detected
J = the reported value is an estimated concentration
UJ = the compound was not detected; the associated reporting limit is approximate
ND = Non-detect

SA = Sample
DU = Duplicate

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		
Loc ID									MW25-2		MW25-2		MW25-2		MW25-2		MW25-2		MW25-2		MW25-2		MW25-2		MW25-2		MW25-2		
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GR
Sample ID									25LM20000		25LM20010		25LM20014		25LM20020		25LM20031		25LM20042		25LM20048		25LM20053		25LM20054		25LM20064		25LM20071		
Sample Depth Interval (FT)									0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		0-0		0-0		
Sample Date									4/12/2006		8/9/2006		8/9/2006		6/6/2007		3/4/2008		4/29/2009		4/29/2009		1/11/2010		1/11/2010		8/3/2010		8/3/2010		
QC Type									SA		SA		DU		SA		SA		SA		DU		SA		DU		SA		DU		
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		
Sample Round									1		2		2		3		4		5		5		6		6		7		7		
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	
Volatile Organic Compounds																															
1,1,1-Trichloroethane	UG/L	0.62	2%	GA	5	0	2	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	3.2	U	3.2	U	1.6	U	1.6	U	
1,1,2,2-Tetrachloroethane	UG/L	0	0%	GA	5	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	0.9	U	0.9	U	0.45	U	0.45	U	
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/L	0	0%	GA	5	0	0	102	5	U	1	U	1	U			1	U	1	U	1	U	4	U	4	U	2	U	2	U	
1,1,2-Trichloroethane	UG/L	0	0%	GA	1	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	2	U	2	U	1	U	1	U	
1,1-Dichloroethane	UG/L	3.5	9%	GA	5	0	10	107	5	U	1	U	1	U	1	UJ	1	U	1.3		1.4		1.5	U	1.5	U	3.5	J	2.8	J	
1,1-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	3.7	U	3.7	U	1.9	U	1.9	U	
1,2,4-Trichlorobenzene	UG/L	0	0%	GA	5	0	0	102	5	UJ	1	U	1	U			1	U	1	U	1	U	1.9	U	1.9	U	0.95	U	0.95	U	
1,2,4-Trimethylbenzene	UG/L	0.45	7%	GA	5	0	2	30			1	U	1	U																	
1,2-Dibromo-3-chloropropane	UG/L	0	0%	GA	0.04	0	0	102	5	UJ	1	U	1	U			2	U	2	U	2	U	4.3	U	4.3	U	2.2	U	2.2	U	
1,2-Dibromoethane	UG/L	0	0%	GA	0.0006	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	1.8	U	1.8	U	0.9	U	0.9	U	
1,2-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	5	U	1	U	1	U			1	U	1	U	1	U	4	U	4	U	2	U	2	U	
1,2-Dichloroethane	UG/L	0.49	3%	GA	0.6	0	3	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	1.5	U	1.5	U	0.71	U	0.71	U	
1,2-Dichloroethene (total)	UG/L	15	15%	GA	5	2	3	20																							
1,2-Dichloropropane	UG/L	0	0%	GA	1	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	1.5	U	1.5	U	0.75	U	0.75	U	
1,3,5-Trimethylbenzene	UG/L	0	0%	GA	5	0	0	30			1	U	1	U																	
1,3-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	5	U	1	U	1	U			1	U	1	U	1	U	3.6	U	3.6	U	1.8	U	1.8	U	
1,4-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	5	U	1	U	1	U			1	U	1	U	1	U	3.5	U	3.5	U	1.8	U	1.8	U	
Acetone	UG/L	11	7%				7	107	25	U	10	UJ	7.6	UJ	5	UJ	10	UJ	35	UJ	20	UJ	50	U	50	U	8	U	8	U	
Benzene	UG/L	62	29%	GA	1	19	31	107	**16**		**2**		**2.2**		**15**	**J**	0.51	J	**17**		**20**		1.8	U	**4**	**J**	**62**	**J**	**57**	**J**	
Bromodichloromethane	UG/L	0	0%	MCL	80	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	1.8	U	1.8	U	0.86	U	0.86	U	
Bromoform	UG/L	0	0%	MCL	80	0	0	107	5	U	1	U	1	U	2	UJ	1	UJ	1	U	1	U	2	U	2	U	1	U	1	U	
Carbon disulfide	UG/L	0.61	2%				2	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	3.6	UJ	3.6	U	1.8	U	1.8	U	
Carbon tetrachloride	UG/L	0	0%	GA	5	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	3.6	U	3.6	U	1.8	U	1.8	U	
Chlorobenzene	UG/L	0	0%	GA	5	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	2.6	U	2.6	U	1.3	U	1.3	U	
Chlorodibromomethane	UG/L	0	0%	MCL	80	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	1.1	U	1.1	U	0.55	U	0.55	U	
Chloroethane	UG/L	0.67	2%	GA	5	0	2	107	5	UJ	1	UJ	1	UJ	1	UJ	2	U	0.51	J	0.67	J	2.1	U	2.1	U	1.1	U	1.1	U	
Chloroform	UG/L	0.32	1%	GA	7	0	1	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	1.6	U	1.6	U	0.8	U	0.8	U	
Cis-1,2-Dichloroethene	UG/L	19	14%	GA	5	4	15	107	5	U	1	U	1	U	1.5	J	1	U	3.6		3.6		2	J	2.8	J	**19**	**J**	**16**	**J**	
Cis-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	1.5	U	1.5	U	0.71	U	0.71	U	
Cyclohexane	UG/L	8.6	11%				11	102	8.6		1	U	1	U			1	U	6.7	J	4.8	J	1.5	U	1.5	U	2.1	J	2.2	J	
Dichlorodifluoromethane	UG/L	0	0%	GA	5	0	0	82	5	U	1	U	1	U			1	UJ	1	U	1	U	1.8	U	1.8	U	0.9	U	0.9	U	
Diisoproply Ether	UG/L	0	0%				0	20																							
Ethyl benzene	UG/L	26	16%	GA	5	12	17	107	**19**		0.77	J	0.98	J	**12**	**J**	0.67	J	**11**		**11**		4.2	U	4.2	U	**26**	**J**	**25**	**J**	
Isopropylbenzene	UG/L	2.6	9%	GA	5	0	10	107	5	U	1	U	1	U	0.45	J	1	U	1.8	J	1.3	J	3.5	U	3.5	U	1.8	U	1.8	U	
Meta/Para Xylene	UG/L	19	11%	GA	5	4	7	61							1.9	J	0.71	J	2	U	2	U	8.2	U	8.2	U	**18**		**19**		
Methyl Acetate	UG/L	0	0%				0	102	5	UJ	1	U	1	U			10	U	2	U	2	U	4.7	U	4.7	U	2.4	U	2.4	U	
Methyl bromide	UG/L	0	0%	GA	5	0	0	107	5	U	1	U	1	U	1	U	2	U	1	UJ	1	UJ	4	U	4	U	2	U	2	U	
Methyl butyl ketone	UG/L	1.9	1%				1	107	25	U	5	U	5	U	2	U	5	U	5	U	5	U	4	U	4	U	2	U	2	U	
Methyl chloride	UG/L	0	0%	GA	5	0	0	107	5	U	1	U	1	U	1	UJ	2	U	1	U	1	U	1.8	U	1.8	U	0.9	U	0.9	U	
Methyl cyclohexane	UG/L	4.2	7%				7	102	4.2	J	1	U	1	U			1	U	3.9	J	2	J	1.6	U	1.6	U	0.8	U	0.8	U	
Methyl ethyl ketone	UG/L	9	10%				11	107	25	U	5	UJ	5	UJ	2	UJ	0.59	J	9	J	4.3	J	10	U	10	U	5	U	5	U	
Methyl isobutyl ketone	UG/L	0	0%				0	107	25	U	5	U	5	U	1	UJ	5	U	5	U	5	U	3.5	U	3.5	U	1.8	U	1.8	U	
Methyl Tertbutyl Ether	UG/L	0	0%				0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	1.3	U	1.3	U	0.65	U	0.65	U	
Methylene chloride	UG/L	0	0%	GA	5	0	0	107	5	U	1	UJ	1	UJ	1	UJ	1	U	1	U	1	U	1.3	U	1.3	U	0.65	U	0.65	U	
Naphthalene	UG/L	0.23	4%				1	25							0.23	J															
n-Butylbenzene	UG/L	0	0%	GA	5	0	0	20																							
Ortho Xylene	UG/L	6.4	8%	GA	5	3	5	61							1	UJ	1	U	1	U	1	U	4	U	4	U	**5.3**		**6.4**		
p-Isopropyltoluene	UG/L	0	0%	GA	5	0	0	10			1	U	1	U																	
Propylbenzene	UG/L	0	0%	GA	5	0	0	10			1	U	1	U																	
sec-Butylbenzene	UG/L	0	0%	GA	5	0	0	20																							
Styrene	UG/L	0	0%	GA	5	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	UJ	1	U	3.6	U	3.6	U	1.8	U	1.8	U	
tert-Butylbenzene	UG/L	0	0%	GA	5	0	0	20																							
Tetrachloroethene	UG/L	0	0%	GA	5	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	4.2	U	4.2	U	2.1	U	2.1	U	
Toluene	UG/L	14	11%	GA	5	5	12	107	5	U	1	U	1	U	1.6	U	1	U	1.2		1.3		2.1	U	2.1	U	**6.1**		**6.5**		
Total Xylenes	UG/L	62	11%	GA	5	3	7	66	15	U	3	U	3	U																	
Trans-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	1.6	U	1.6	U	0.8	U	0.8	U	
Trans-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	1.8	U	1.8	U	0.86	U	0.86	U	
Trichloroethene	UG/L	2	10%	GA	5	0	11	107	5	U	1	U	1	U	0.51	J	1	U	1	U	1	U	1.9	U	1.9	U	1.6	J	1.6	J	
Trichlorofluoromethane	UG/L	0	0%	GA	5	0	0	102	5	U	1	U	1	U			1	U	1	U	1	U	1.6	U	1.6	U	0.8	U	0.8	U	
Vinyl chloride	UG/L	2.6	6%	GA	2	2	6	107	5	U	1	U	1	U	1	UJ	1	U	1	U	1	U	2.2	U	2.2	U	**2.6**	**J**	**2.4**	**J**	

Area									SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25
Loc ID									MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-2
Matrix									GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER
Sample ID									25LM20000	25LM20010	25LM20014	25LM20020	25LM20031	25LM20042	25LM20048	25LM20053	25LM20054	25LM20064	25LM20071
Sample Depth Interval (FT)									0-0.1	0-0.1	0-0.1	0-0.1	0-0.1	0-0.1	0-0.1	0-0.1	0-0.1	0-0	0-0
Sample Date									4/12/2006	8/9/2006	8/9/2006	6/6/2007	3/4/2008	4/29/2009	4/29/2009	1/11/2010	1/11/2010	8/3/2010	8/3/2010
QC Type									SA	SA	DU	SA	SA	SA	DU	SA	DU	SA	DU
Study ID									LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM
Sample Round									1	2	2	3	4	5	5	6	6	7	7
Filtered									Total	Total	Total	Total	Total	Total	Total	Total	Total	Total	Total
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual
Semivolatile Organic Compounds																			
1,1'-Biphenyl	UG/L	0	0%	GA	5	0	0	18	10 U	10 U	10 U								
2,4,5-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18	10 U	10 U	10 U								
2,4,6-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18	10 U	10 U	10 U								
2,4-Dichlorophenol	UG/L	0	0%	GA	5	0	0	18	10 U	10 U	10 U								
2,4-Dimethylphenol	UG/L	0	0%				0	18	10 U	10 U	10 U								
2,4-Dinitrophenol	UG/L	0	0%				0	18	49 U	48 U	49 U								
2,4-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18	10 U	10 U	10 U								
2,6-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18	10 U	10 U	10 U								
2-Chloronaphthalene	UG/L	0	0%				0	18	10 U	10 U	10 U								
2-Chlorophenol	UG/L	0	0%				0	18	10 U	10 U	10 U								
2-Methylnaphthalene	UG/L	0	0%				0	18	10 U	10 U	10 U								
2-Methylphenol	UG/L	0	0%				0	18	10 U	10 U	10 U								
2-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	49 U	48 U	49 U								
2-Nitrophenol	UG/L	0	0%	GA	1	0	0	18	10 U	10 U	10 U								
3,3'-Dichlorobenzidine	UG/L	0	0%	GA	5	0	0	18	20 U	19 U	20 U								
3-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	49 U	48 U	49 U								
4,6-Dinitro-2-methylphenol	UG/L	0	0%	GA	1	0	0	18	49 U	48 U	49 U								
4-Bromophenyl phenyl ether	UG/L	0	0%				0	18	10 U	10 U	10 U								
4-Chloro-3-methylphenol	UG/L	0	0%	GA	1	0	0	18	10 U	10 U	10 U								
4-Chloroaniline	UG/L	0	0%	GA	5	0	0	18	10 U	10 U	10 U								
4-Chlorophenyl phenyl ether	UG/L	0	0%				0	18	10 U	10 U	10 U								
4-Methylphenol	UG/L	0	0%				0	18	10 U	10 U	10 U								
4-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	49 U	48 U	49 U								
4-Nitrophenol	UG/L	0	0%	GA	1	0	0	18	49 U	48 U	49 U								
Acenaphthene	UG/L	0.5	6%				1	18	10 U	10 U	10 U								
Acenaphthylene	UG/L	2	22%				4	18	10 U	10 U	10 U								
Acetophenone	UG/L	0	0%				0	18	10 U	10 U	10 U								
Anthracene	UG/L	1	6%				1	18	10 U	10 U	10 U								
Atrazine	UG/L	0	0%	GA	7.5	0	0	18	10 U	10 U	10 U								
Benzaldehyde	UG/L	0	0%				0	18	49 U	48 U	49 U								
Benzo(a)anthracene	UG/L	0	0%				0	18	10 U	10 U	10 U								
Benzo(a)pyrene	UG/L	0	0%	GA	0		0	18	10 U	10 U	10 U								
Benzo(b)fluoranthene	UG/L	0	0%				0	18	10 U	10 U	10 U								
Benzo(ghi)perylene	UG/L	0.6	6%				1	18	10 U	10 U	10 U								
Benzo(k)fluoranthene	UG/L	0	0%				0	18	10 U	10 U	10 U								
Bis(2-Chloroethoxy)methane	UG/L	0	0%	GA	5	0	0	18	10 U	10 U	10 U								
Bis(2-Chloroethyl)ether	UG/L	0	0%	GA	1	0	0	18	10 U	10 U	10 U								
Bis(2-Chloroisopropyl)ether	UG/L	0	0%	GA	5	0	0	18	10 U	10 U	10 U								
Bis(2-Ethylhexyl)phthalate	UG/L	11	6%	GA	5	1	1	18	10 U	10 U	10 U								
Butylbenzylphthalate	UG/L	2	6%				1	18	10 U	10 U	10 U								
Caprolactam	UG/L	0	0%				0	18	10 U	10 U	10 U								
Carbazole	UG/L	0	0%				0	18	10 U	10 U	10 U								
Chrysene	UG/L	0	0%				0	18	10 U	10 U	10 U								
Dibenz(a,h)anthracene	UG/L	0	0%				0	18	10 U	10 U	10 U								
Dibenzofuran	UG/L	0	0%				0	18	10 U	10 U	10 U								
Diethyl phthalate	UG/L	0	0%				0	18	10 U	10 U	10 U								
Dimethylphthalate	UG/L	0	0%				0	18	10 U	10 U	10 U								
Di-n-butylphthalate	UG/L	0	0%	GA	50	0	0	18	10 U	10 U	10 U								
Di-n-octylphthalate	UG/L	0	0%				0	18	10 U	10 U	10 U								
Fluoranthene	UG/L	0	0%				0	18	10 U	10 U	10 U								
Fluorene	UG/L	0	0%				0	18	10 U	10 UJ	10 UJ								
Hexachlorobenzene	UG/L	0	0%	GA	0.04	0	0	18	10 U	10 U	10 U								
Hexachlorobutadiene	UG/L	0	0%	GA	0.5	0	0	18	10 U	10 U	10 U								
Hexachlorocyclopentadiene	UG/L	0	0%	GA	5	0	0	18	44 U	43 U	44 U								
Hexachloroethane	UG/L	0	0%	GA	5	0	0	18	10 U	10 U	10 U								
Indeno(1,2,3-cd)pyrene	UG/L	0	0%				0	18	10 U	10 U	10 U								
Isophorone	UG/L	0	0%				0	18	10 U	10 U	10 U								
Naphthalene	UG/L	2	6%				1	18	10 U	10 U	10 U								
Nitrobenzene	UG/L	0	0%	GA	0.4	0	0	18	10 U	10 U	10 U								
N-Nitroso-di-n-propylamine	UG/L	0	0%				0	18	10 U	10 U	10 U								
N-Nitrosodiphenylamine	UG/L	0	0%				0	18	10 U	10 U	10 U								
Pentachlorophenol	UG/L	0	0%	GA	1	0	0	18	49 U	48 U	49 U								
Phenanthrene	UG/L	0	0%				0	18	10 U	10 U	10 U								
Phenol	UG/L	0	0%	GA	1	0	0	18	10 U	10 U	10 U								
Pyrene	UG/L	0	0%				0	18	10 U	10 U	10 U								

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Area	Loc ID	Matrix	Sample ID	Sample Depth Interval (FT)	Sample Date	QC Type	Study ID	Sample Round	Filtered	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
									SEAD-25	SEAD-25	GROUNDWATER	25LM20000	0-0.1	4/12/2006	SA	LTM	1	Total																				
									SEAD-25	MW25-2	GROUNDWATER	25LM20010	0-0.1	8/9/2006	SA	LTM	2	Total																				
									SEAD-25	MW25-2	GROUNDWATER	25LM20014	0-0.1	8/9/2006	DU	LTM	2	Total																				
									SEAD-25	MW25-2	GROUNDWATER	25LM20020	0-0.1	6/6/2007	SA	LTM	3	Total																				
									SEAD-25	MW25-2	GROUNDWATER	25LM20031	0-0.1	3/4/2008	SA	LTM	4	Total																				
									SEAD-25	MW25-2	GROUNDWATER	25LM20042	0-0.1	4/29/2009	SA	LTM	5	Total																				
									SEAD-25	MW25-2	GROUNDWATER	25LM20048	0-0.1	4/29/2009	DU	LTM	5	Total																				
									SEAD-25	MW25-2	GROUNDWATER	25LM20053	0-0.1	1/11/2010	SA	LTM	6	Total																				
									SEAD-25	MW25-2	GROUNDWATER	25LM20054	0-0.1	1/11/2010	DU	LTM	6	Total																				
									SEAD-25	MW25-2	GROUNDWATER	25LM20064	0-0	8/3/2010	SA	LTM	7	Total																				
									SEAD-25	MW25-2	GROUNDWATER	25LM20071	0-0	8/3/2010	DU	LTM	7	Total																				
Inorganics																																				
Iron	UG/L	15,700	85%	GA	300	56	84	99											**2,510**	**J**	**606**		**727**		**2,600**	**J**	**711**		**14,400**		**15,700**		**2,900**		**2,410**		**1,660**	
Sodium	UG/L	58,100	100%	GA	20,000	14	99	99											4,730		5,690	J	5,510	J	6,000	J	3,460		7,100		7,100		7,880		7,720		10,300	
Wet Chemistry - MEE																																				
Chloride	MG/L	97.9	78%	GA	250	0	78	100											6.5		2.2	J	2.2	J	4		0.2	U	2.2		2.2		0.5	U	2.8		3		2.8	
Ethane	UG/L	1.1	5%				5	104											2	U	10	U	10	U	0.24		1	U	1	U	1	U	0.16	U	0.16	U	0.16	U	0.16	U
Ethene	UG/L	4.6	5%				5	104											2	U	10	U	10	U	4.2		1	U	1	U	1	U	0.17	U	0.17	U	0.17	U	0.17	U
Methane	UG/L	170	57%				59	104											80	J	36		35		170		3.2	J	68		64		20		22		120		130	
Nitrate	MG/L	6.4	68%	GA	10	0	45	66																	0.5	J			0.05	U	0.05	U	0.199	J	0.05	UJ				
Nitrate Nitrogen	MG/L	1	45%				13	29											0.05	U	0.05	U	0.05	U			0.305	J												
Nitrate/Nitrite Nitrogen	MG/L	1	64%	GA	10	0	16	25																			0.305						0.199	J	0.003	UJ	0.013	UJ	0.013	UJ
Nitrite	MG/L	0.73	27%	GA	1	0	18	66																	0.5				0.01	U	0.01	U	0.007	UJ	0.007	UJ				
Nitrite Nitrogen	MG/L	0.087	3%				1	29											0.05	U	0.05	U	0.05	U			0.01	UJ												
Sulfate	MG/L	182	100%	GA	250	0	100	100											39.6		33.2		31		22		31.1		75.8		82.6		64.4	J	64.8	J	40.3		45.3	
Field Measurement - Hach Kit																																				
Conductivity	S/m	1.26	100%				90	90											0.551		0.562		0.562		0.454		0.64		0.702		0.702		0.573		0.573		1.09		1.09	
Conductivity (post)	S/m	0.844	100%				3	3																																
Conductivity (pre)	S/m	0.83	100%				3	3																																
Dissolved Oxygen	MG/L	12.6	100%				86	86											6.29		0.3		0.3		0.07		1.35		0.11		0.11		0.41		0.41		0.02		0.02	
Dissolved Oxygen (post)	MG/L	5.17	100%				3	3																																
Dissolved Oxygen (pre)	MG/L	5.36	100%				3	3																																
Nitrate Nitrogen	MG/L	0.5	100%				3	3																																
Nitrite Nitrogen	MG/L	0.5	100%				3	3																																
ORP	mV	259	100%				90	90											-11		-82		-82		-92		-60		-115		-115		-151		-151		-230		-230	
ORP (post)	mV	197	100%				3	3																																
ORP (pre)	mV	193	100%				3	3																																
pH	Std units	8.37	100%				90	90											7.17		6.93		6.93		7.11		7.15		6.84		6.84		7.25		7.25		6.79		6.79	
pH (post)	Std units	7.38	100%				3	3																																
pH (pre)	Std units	7.38	100%				3	3																																
Sulfide	MG/L	1.04	89%				72	81											0.01		0.15		0.15				0.01	U	0.04		0.04		0.16		0.16					
Temperature	DEG C	26.55	100%				93	93											10.5		26.55		26.55		12.4		3.2		8.1		8.1		6.3		6.3		21.2		21.2	
Turbidity	NTU	195	100%				90	90											16.1		2.3		2.3		11		2.78		0.9		0.9		1.06		1.06		3.4		3.4	
Turbidity (post)	NTU	7.6	100%				2	2																																
Turbidity (pre)	NTU	5.7	100%				1	1																																

Notes:
1. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
2. Shading indicates concentration above cleanup goal.

U = compound was not detected
J = the reported value is an estimated concentration
UJ = the compound was not detected; the associated reporting limit is approximate
ND = Non-detect

SA = Sample
DU = Duplicate

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-25 MW25-2 GROUNDWATER 25LM20079 10.25-10.25 2/8/2011 SA LTM 8 Total	Qual	SEAD-25 MW25-2 GROUNDWATER 25LM20080 10.25-10.25 2/8/2011 DU LTM 8 Total	Qual	SEAD-25 MW25-2 GROUNDWATER 25LM20091 10.26-10.26 3/1/2012 SA LTM 9 Total	Qual	SEAD-25 MW25-2 GROUNDWATER 25LM20101 10.25-10.25 5/8/2013 SA LTM 10 Total	Qual	SEAD-25 MW25-2 GROUNDWATER 25LM20102 10.25-10.25 5/8/2013 DU LTM 10 Total	Qual	SEAD-25 MW25-2 GROUNDWATER 25LM20109 10.26-10.26 6/18/2014 SA LTM 11 Total	Qual	SEAD-25 MW25-2 GROUNDWATER 25LM20113 10.25-10.25 3/18/2015 SA LTM 12 Total	Qual	SEAD-25 MW25-2 GROUNDWATER 25LM20119 10-10 3/17/2016 SA LTM 13 Total	Qual	SEAD-25 MW25-2 GROUNDWATER 25LM20120 10-10 3/17/2016 DU LTM 13 Total	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20001 0-0.1 1/31/2006 DU LTM 1 Total	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20002 0-0.1 1/31/2006 SA LTM 1 Total	Qual	GR
Volatile Organic Compounds																																		
1,1,1-Trichloroethane	UG/L	0.62	2%	GA	5	0	2	107	0.2	UJ	0.2	UJ	0.2	U	0.5	U	0.5	U	0.5	U	0.37	U	0.37	U	0.37	U	1	U	1	U		
1,1,2,2-Tetrachloroethane	UG/L	0	0%	GA	5	0	0	107	0.38	UJ	0.38	UJ	0.38	U	0.18	U	0.18	U	0.18	U	0.62	U	0.62	U	0.62	U	1	U	1	U		
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/L	0	0%	GA	5	0	0	102	0.31	U	0.31	U	0.31	UJ	0.5	U	0.5	U	0.5	U	0.36	U	0.36	U	0.36	U	1	U	1	U		
1,1,2-Trichloroethane	UG/L	0	0%	GA	1	0	0	107	0.33	UJ	0.33	UJ	0.33	U	0.13	U	0.13	U	0.13	U	0.33	U	0.33	U	0.33	U	1	U	1	U		
1,1-Dichloroethane	UG/L	3.5	9%	GA	5	0	10	107	1.5	J	1.4	J	0.21	U	1.2		1.4		1.8		0.38	U	0.38	U	0.38	U	1	U	1	U		
1,1-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	0.35	UJ	0.35	UJ	0.35	U	0.11	U	0.11	U	0.11	U	0.36	U	0.36	U	0.36	U	1	U	1	U		
1,2,4-Trichlorobenzene	UG/L	0	0%	GA	5	0	0	102	0.37	UJ	0.37	UJ	0.37	U	0.25	UJ	0.25	UJ	0.25	UJ	2.5	U	2.5	U	2.5	U	1	U	1	U		
1,2,4-Trimethylbenzene	UG/L	0.45	7%	GA	5	0	2	30	0.4	J	0.45	J	0.19	U																		
1,2-Dibromo-3-chloropropane	UG/L	0	0%	GA	0.04	0	0	102	0.5	U	0.5	U	0.5	U	0.44	U	0.44	U	0.44	U	1.1	U	1.1	U	1.1	U	1	U	1	U		
1,2-Dibromoethane	UG/L	0	0%	GA	0.0006	0	0	107	0.22	UJ	0.22	UJ	0.22	U	0.25	U	0.25	U	0.25	U	0.44	U	0.44	U	0.44	U	1	U	1	U		
1,2-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.15	UJ	0.15	UJ	0.15	U	0.21	U	0.21	U	0.21	U	0.37	U	0.37	U	0.37	U	1	U	1	U		
1,2-Dichloroethane	UG/L	0.49	3%	GA	0.6	0	3	107	0.2	UJ	0.2	UJ	0.2	U	0.32	J	0.1	U	0.44	J	0.5	U	0.5	U	0.5	U	1	U	1	U		
1,2-Dichloroethene (total)	UG/L	15	15%	GA	5	2	3	20	**15**	**J**	**11**	**J**	0.76	J																		
1,2-Dichloropropane	UG/L	0	0%	GA	1	0	0	107	0.25	UJ	0.25	UJ	0.25	U	0.13	U	0.13	U	0.13	U	0.67	U	0.67	U	0.67	U	1	U	1	U		
1,3,5-Trimethylbenzene	UG/L	0	0%	GA	5	0	0	30	0.2	UJ	0.2	UJ	0.2	U																		
1,3-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.26	UJ	0.26	UJ	0.26	U	0.25	U	0.25	U	0.25	U	0.43	U	0.43	U	0.43	U	1	U	1	U		
1,4-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.24	UJ	0.24	UJ	0.24	U	0.28	U	0.28	U	0.28	U	0.46	U	0.46	U	0.46	U	1	U	1	U		
Acetone	UG/L	11	7%				7	107	9.5		8.6		2.2	U	11	J	8.7	J	6.9	J	7	U	7	U	7	U	5	U	5	U		
Benzene	UG/L	62	29%	GA	1	19	31	107	**14**	**J**	**12**	**J**	0.99	J	**19**		**20**		**24**		0.64	J	1		**1.1**		1	U	1	U		
Bromodichloromethane	UG/L	0	0%	MCL	80	0	0	107	0.33	UJ	0.33	UJ	0.33	U	0.25	U	0.25	U	0.25	U	0.44	U	0.44	U	0.44	U	1	U	1	U		
Bromoform	UG/L	0	0%	MCL	80	0	0	107	0.23	UJ	0.23	UJ	0.23	U	0.5	UJ	0.5	UJ	0.5	U	0.43	U	0.43	UJ	0.43	UJ	1	U	1	U		
Carbon disulfide	UG/L	0.61	2%				2	107	0.61	J	0.56	J	0.25	U	0.6	U	0.6	U	0.6	U	1	U	1	U	1	U	1	U	1	U		
Carbon tetrachloride	UG/L	0	0%	GA	5	0	0	107	0.22	UJ	0.22	UJ	0.22	U	0.5	UJ	0.5	UJ	0.5	U	0.33	U	0.33	U	0.33	U	1	U	1	U		
Chlorobenzene	UG/L	0	0%	GA	5	0	0	107	0.22	UJ	0.22	UJ	0.22	U	0.25	U	0.25	U	0.25	U	0.26	U	0.26	U	0.26	U	1	U	1	U		
Chlorodibromomethane	UG/L	0	0%	MCL	80	0	0	107	0.3	UJ	0.3	UJ	0.3	U	0.1	UJ	0.1	UJ	0.1	U	0.32	U	0.32	U	0.32	U	1	U	1	U		
Chloroethane	UG/L	0.67	2%	GA	5	0	2	107	0.55	U	0.55	U	0.55	U	2	UJ	2	UJ	2	U	2.5	U	2.5	U	2.5	U	1	U	1	U		
Chloroform	UG/L	0.32	1%	GA	7	0	1	107	0.32	J	0.32	UJ	0.32	U	0.14	U	0.14	U	0.14	U	0.5	U	0.5	U	0.5	U	1	U	1	U		
Cis-1,2-Dichloroethene	UG/L	19	14%	GA	5	4	15	107	**15**	**J**	**11**	**J**	0.76	J	2.8		2.9		3.5		0.41	U	0.41	U	0.41	U	1	U	1	U		
Cis-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	0.19	UJ	0.19	UJ	0.19	UJ	0.11	U	0.11	U	0.11	U	0.4	U	0.4	U	0.4	U	1	U	1	U		
Cyclohexane	UG/L	8.6	11%				11	102	1.9	J	1.8	J	0.31	U	1.7		1.8		3		0.39	U	0.39	U	0.39	U	1	U	1	U		
Dichlorodifluoromethane	UG/L	0	0%	GA	5	0	0	82							0.25	U	0.25	U	0.25	U	0.6	U	0.6	U	0.6	U	1	UJ	1	UJ		
Diisoproply Ether	UG/L	0	0%				0	20	0.21	U	0.21	U	0.21	U																		
Ethyl benzene	UG/L	26	16%	GA	5	12	17	107	**8.1**	**J**	**6.5**	**J**	0.47	J	**10**		**11**		**14**		0.33	U	0.33	U	0.33	U	1	U	1	U		
Isopropylbenzene	UG/L	2.6	9%	GA	5	0	10	107	0.49	J	0.4	J	0.23	U	1		1.1		1.2		0.35	U	0.35	U	0.35	U	1	U	1	U		
Meta/Para Xylene	UG/L	19	11%	GA	5	4	7	61	**6.5**	**J**	**5.1**	**J**	0.59	U																		
Methyl Acetate	UG/L	0	0%				0	102	0.53	U	0.53	U	0.53	U	0.19	UJ	0.19	UJ	0.19	U	1.8	U	1.8	U	1.8	U	1	U	1	U		
Methyl bromide	UG/L	0	0%	GA	5	0	0	107	0.49	U	0.49	U	0.49	UJ	2	U	2	U	2	U	2.5	U	2.5	UJ	2.5	UJ	1	U	1	U		
Methyl butyl ketone	UG/L	1.9	1%				1	107	1.7	U	1.7	U	1.7	U	1.9	J	1	U	1	U	2	U	2	U	2	U	5	U	5	U		
Methyl chloride	UG/L	0	0%	GA	5	0	0	107	0.36	U	0.36	U	0.36	U	0.33	U	0.33	U	0.33	U	0.4	U	0.4	U	0.4	U	1	U	1	U		
Methyl cyclohexane	UG/L	4.2	7%				7	102	0.52	J	0.46	J	0.3	U	0.1	U	0.1	U	0.41	J	0.43	U	0.43	U	0.43	U	1	U	1	U		
Methyl ethyl ketone	UG/L	9	10%				11	107	3.2	J	2.3	J	1.3	U	3.8	J	2.9	J	1	U	3.4	U	3.4	U	3.4	U	5	U	5	U		
Methyl isobutyl ketone	UG/L	0	0%				0	107	1.3	U	1.3	U	1.3	U	1	U	1	U	1	U	2.1	U	2.1	U	2.1	U	5	U	5	U		
Methyl Tertbutyl Ether	UG/L	0	0%				0	107	0.36	U	0.36	U	0.36	U	0.2	U	0.2	U	0.2	U	0.3	U	0.3	U	0.3	U	1	U	1	U		
Methylene chloride	UG/L	0	0%	GA	5	0	0	107	1.1	U	1.1	U	1.1	U	1	U	1	U	1	U	2.5	U	2.5	U	2.5	U	1	U	1	U		
Naphthalene	UG/L	0.23	4%				1	25	0.3	UJ	0.3	UJ	0.3	U																		
n-Butylbenzene	UG/L	0	0%	GA	5	0	0	20	0.23	UJ	0.23	UJ	0.23	U																		
Ortho Xylene	UG/L	6.4	8%	GA	5	3	5	61	**5.6**	**J**	4.3	J	0.25	U																		
p-Isopropyltoluene	UG/L	0	0%	GA	5	0	0	10																								
Propylbenzene	UG/L	0	0%	GA	5	0	0	10																								
sec-Butylbenzene	UG/L	0	0%	GA	5	0	0	20	0.21	UJ	0.21	UJ	0.21	U																		
Styrene	UG/L	0	0%	GA	5	0	0	107	0.23	UJ	0.23	UJ	0.23	U	0.11	U	0.11	U	0.11	U	0.27	U	0.27	U	0.27	U	1	U	1	U		
tert-Butylbenzene	UG/L	0	0%	GA	5	0	0	20	0.31	UJ	0.31	UJ	0.31	U																		
Tetrachloroethene	UG/L	0	0%	GA	5	0	0	107	0.4	U	0.4	U	0.4	U	0.15	U	0.15	U	0.15	U	0.74	U	0.74	UJ	0.74	UJ	1	U	1	U		
Toluene	UG/L	14	11%	GA	5	5	12	107	**7.4**	**J**	**5.4**	**J**	0.27	U	0.91	J	0.87	J	2		0.48	U	0.48	U	0.48	U	1	U	1	U		
Total Xylenes	UG/L	62	11%	GA	5	3	7	66	**12**	**J**	**9.4**	**J**	0.25	U	1	J	1	J	2.2		0.23	U	0.23	U	0.23	U	3	U	3	U		
Trans-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	0.25	U	0.25	U	0.25	U	0.2	U	0.2	U	0.2	U	0.37	U	0.37	U	0.37	U	1	U	1	U		
Trans-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	0.2	UJ	0.2	UJ	0.2	UJ	0.21	U	0.21	U	0.21	U	0.42	U	0.42	U	0.42	U	1	U	1	U		
Trichloroethene	UG/L	2	10%	GA	5	0	11	107	2	J	1.8	J	0.45	J	1.7		1.7		0.88	J	0.48	U	0.48	U	0.48	U	1	U	1	U		
Trichlorofluoromethane	UG/L	0	0%	GA	5	0	0	102	0.24	U	0.24	U	0.24	U	0.25	U	0.25	U	0.25	U	0.42	U	0.42	U	0.42	U	1	UJ	1	UJ		
Vinyl chloride	UG/L	2.6	6%	GA	2	2	6	107	0.79	J	0.25	U	0.25	U	0.66	J	0.67	J	1.3		0.5	U	0.5	U	0.5	U	1	U	1	U		

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Area	Loc ID	Matrix	Sample ID	Sample Depth Interval (FT)	Sample Date	QC Type	Study ID	Sample Round	Filtered	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
									SEAD-25	SEAD-25	GROUNDWATER	25LM20079	10.25-10.25	2/8/2011	SA	LTM	8	Total																		
									SEAD-25	MW25-2	GROUNDWATER	25LM20080	10.25-10.25	2/8/2011	DU	LTM	8	Total																		
									SEAD-25	MW25-2	GROUNDWATER	25LM20091	10.26-10.26	3/1/2012	SA	LTM	9	Total																		
									SEAD-25	MW25-2	GROUNDWATER	25LM20101	10.25-10.25	5/8/2013	SA	LTM	10	Total																		
									SEAD-25	MW25-2	GROUNDWATER	25LM20102	10.25-10.25	5/8/2013	DU	LTM	10	Total																		
									SEAD-25	MW25-2	GROUNDWATER	25LM20109	10.26-10.26	6/18/2014	SA	LTM	11	Total																		
									SEAD-25	MW25-2	GROUNDWATER	25LM20113	10.25-10.25	3/18/2015	SA	LTM	12	Total																		
									SEAD-25	MW25-2	GROUNDWATER	25LM20119	10-10	3/17/2016	SA	LTM	13	Total																		
									SEAD-25	MW25-2	GROUNDWATER	25LM20120	10-10	3/17/2016	DU	LTM	13	Total																		
									SEAD-25	MW25-3	GROUNDWATER	25LM20001	0-0.1	1/31/2006	DU	LTM	1	Total																		
									SEAD-25	MW25-3	GROUNDWATER	25LM20002	0-0.1	1/31/2006	SA	LTM	1	Total																		
Semivolatile Organic Compounds																																			
1,1'-Biphenyl	UG/L	0	0%	GA	5	0	0	18																									9	U	10	U
2,4,5-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18																									9	U	10	U
2,4,6-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18																									9	U	10	U
2,4-Dichlorophenol	UG/L	0	0%	GA	5	0	0	18																									9	U	10	U
2,4-Dimethylphenol	UG/L	0	0%				0	18																									9	U	10	U
2,4-Dinitrophenol	UG/L	0	0%				0	18																									47	U	48	U
2,4-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18																									9	U	10	U
2,6-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18																									9	U	10	U
2-Chloronaphthalene	UG/L	0	0%				0	18																									9	U	10	U
2-Chlorophenol	UG/L	0	0%				0	18																									9	U	10	U
2-Methylnaphthalene	UG/L	0	0%				0	18																									9	U	10	U
2-Methylphenol	UG/L	0	0%				0	18																									9	U	10	U
2-Nitroaniline	UG/L	0	0%	GA	5	0	0	18																									47	U	48	U
2-Nitrophenol	UG/L	0	0%	GA	1	0	0	18																									9	U	10	U
3,3'-Dichlorobenzidine	UG/L	0	0%	GA	5	0	0	18																									19	U	19	U
3-Nitroaniline	UG/L	0	0%	GA	5	0	0	18																									47	U	48	U
4,6-Dinitro-2-methylphenol	UG/L	0	0%	GA	1	0	0	18																									47	U	48	U
4-Bromophenyl phenyl ether	UG/L	0	0%				0	18																									9	U	10	U
4-Chloro-3-methylphenol	UG/L	0	0%	GA	1	0	0	18																									9	U	10	U
4-Chloroaniline	UG/L	0	0%	GA	5	0	0	18																									9	U	10	U
4-Chlorophenyl phenyl ether	UG/L	0	0%				0	18																									9	U	10	U
4-Methylphenol	UG/L	0	0%				0	18																									9	U	10	U
4-Nitroaniline	UG/L	0	0%	GA	5	0	0	18																									47	U	48	U
4-Nitrophenol	UG/L	0	0%	GA	1	0	0	18																									47	UJ	48	UJ
Acenaphthene	UG/L	0.5	6%				1	18																									9	U	10	U
Acenaphthylene	UG/L	2	22%				4	18																									9	U	10	U
Acetophenone	UG/L	0	0%				0	18																									9	U	10	U
Anthracene	UG/L	1	6%				1	18																									9	U	10	U
Atrazine	UG/L	0	0%	GA	7.5	0	0	18																									9	U	10	U
Benzaldehyde	UG/L	0	0%				0	18																									47	U	48	U
Benzo(a)anthracene	UG/L	0	0%				0	18																									9	U	10	U
Benzo(a)pyrene	UG/L	0	0%	GA	0		0	18																									9	U	10	U
Benzo(b)fluoranthene	UG/L	0	0%				0	18																									9	U	10	U
Benzo(ghi)perylene	UG/L	0.6	6%				1	18																									9	U	10	U
Benzo(k)fluoranthene	UG/L	0	0%				0	18																									9	U	10	U
Bis(2-Chloroethoxy)methane	UG/L	0	0%	GA	5	0	0	18																									9	U	10	U
Bis(2-Chloroethyl)ether	UG/L	0	0%	GA	1	0	0	18																									9	U	10	U
Bis(2-Chloroisopropyl)ether	UG/L	0	0%	GA	5	0	0	18																									9	U	10	U
Bis(2-Ethylhexyl)phthalate	UG/L	11	6%	GA	5	1	1	18																									9	U	10	U
Butylbenzylphthalate	UG/L	2	6%				1	18																									9	U	10	U
Caprolactam	UG/L	0	0%				0	18																									9	U	10	U
Carbazole	UG/L	0	0%				0	18																									9	U	10	U
Chrysene	UG/L	0	0%				0	18																									9	U	10	U
Dibenz(a,h)anthracene	UG/L	0	0%				0	18																									9	U	10	U
Dibenzofuran	UG/L	0	0%				0	18																									9	U	10	U
Diethyl phthalate	UG/L	0	0%				0	18																									9	U	10	U
Dimethylphthalate	UG/L	0	0%				0	18																									9	U	10	U
Di-n-butylphthalate	UG/L	0	0%	GA	50	0	0	18																									9	U	10	U
Di-n-octylphthalate	UG/L	0	0%				0	18																									9	U	10	U
Fluoranthene	UG/L	0	0%				0	18																									9	U	10	U
Fluorene	UG/L	0	0%				0	18																									9	U	10	U
Hexachlorobenzene	UG/L	0	0%	GA	0.04	0	0	18																									9	U	10	U
Hexachlorobutadiene	UG/L	0	0%	GA	0.5	0	0	18																									9	U	10	U
Hexachlorocyclopentadiene	UG/L	0	0%	GA	5	0	0	18																									42	U	44	U
Hexachloroethane	UG/L	0	0%	GA	5	0	0	18																									9	U	10	U
Indeno(1,2,3-cd)pyrene	UG/L	0	0%				0	18																									9	U	10	U
Isophorone	UG/L	0	0%				0	18																									9	U	10	U
Naphthalene	UG/L	2	6%				1	18																									9	U	10	U
Nitrobenzene	UG/L	0	0%	GA	0.4	0	0	18																									9	U	10	U
N-Nitroso-di-n-propylamine	UG/L	0	0%				0	18																									9	U	10	U
N-Nitrosodiphenylamine	UG/L	0	0%				0	18																									9	U	10	U
Pentachlorophenol	UG/L	0	0%	GA	1	0	0	18																									47	U	48	U
Phenanthrene	UG/L	0	0%				0	18																									9	U	10	U
Phenol	UG/L	0	0%	GA	1	0	0	18																									9	U	10	U
Pyrene	UG/L	0	0%				0	18																									9	U	10	U

Area									SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	
Loc ID									MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-2	MW25-3	MW25-3	
Matrix									GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GR
Sample ID									25LM20079	25LM20080	25LM20091	25LM20101	25LM20102	25LM20109	25LM20113	25LM20119	25LM20120	25LM20001	25LM20002	
Sample Depth Interval (FT)									10.25-10.25	10.25-10.25	10.26-10.26	10.25-10.25	10.25-10.25	10.26-10.26	10.25-10.25	10-10	10-10	0-0.1	0-0.1	
Sample Date									2/8/2011	2/8/2011	3/1/2012	5/8/2013	5/8/2013	6/18/2014	3/18/2015	3/17/2016	3/17/2016	1/31/2006	1/31/2006	
QC Type									SA	DU	SA	SA	DU	SA	SA	SA	DU	DU	SA	
Study ID									LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	
Sample Round									8	8	9	10	10	11	12	13	13	1	1	
Filtered									Total	Total	Total	Total	Total	Total	Total	Total	Total	Total	Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	Value Qual	
Inorganics																				
Iron	UG/L	15,700	85%	GA	300	56	84	99	13,100		3,780 J	6,500 J	11,000 J	9,900	340	350	340	86 J	76.4 J	
Sodium	UG/L	58,100	100%	GA	20,000	14	99	99	10,200		9,320 J	16,000 J	10,000 J	9,100	2,500	3,500	3,600	12,300	12,000	
Wet Chemistry - MEE																				
Chloride	MG/L	97.9	78%	GA	250	0	78	100	5.8		0.9 J	1.8 J	1.7 J	1.9	0.74	0.73	0.74	2.1	2.3	
Ethane	UG/L	1.1	5%				5	104	0.58 U	0.58 U	0.58 U	4 U	4 U	0.55 U	0.55 U	0.55 U	0.55 U	2 U	2 U	
Ethene	UG/L	4.6	5%				5	104	0.69 U	0.69 U	0.69 U	3 U	3 U	0.5 U	0.5 U	0.5 U	0.5 U	2 U	2 U	
Methane	UG/L	170	57%				59	104	32	59	31 J	22	25	0.32 J	4.2	4.6	4.1	2 U	2 U	
Nitrate	MG/L	6.4	68%	GA	10	0	45	66	0.0152 U		0.0152 U	0.01 U	0.027 J	0.059	1.3	0.37	0.35			
Nitrate Nitrogen	MG/L	1	45%				13	29										0.05 U	0.05 U	
Nitrate/Nitrite Nitrogen	MG/L	1	64%	GA	10	0	16	25												
Nitrite	MG/L	0.73	27%	GA	1	0	18	66	0.00321 U		0.036 J	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 J			
Nitrite Nitrogen	MG/L	0.087	3%				1	29										0.05 U	0.05 U	
Sulfate	MG/L	182	100%	GA	250	0	100	100	45 J		52 J	160 J	150 J	10	24	36	36	39.9	39.8	
Field Measurement - Hach Kit																				
Conductivity	S/m	1.26	100%				90	90	0.806	0.806	0.681	0.907	0.907		0.411	0.669	0.669	0.49	0.49	
Conductivity (post)	S/m	0.844	100%				3	3												
Conductivity (pre)	S/m	0.83	100%				3	3												
Dissolved Oxygen	MG/L	12.6	100%				86	86	0.24	0.24	0.24	0.11	0.11		8.84	0.61	0.61	1.19	1.19	
Dissolved Oxygen (post)	MG/L	5.17	100%				3	3												
Dissolved Oxygen (pre)	MG/L	5.36	100%				3	3												
Nitrate Nitrogen	MG/L	0.5	100%				3	3												
Nitrite Nitrogen	MG/L	0.5	100%				3	3												
ORP	mV	259	100%				90	90	-148	-148	-106	-350	-350		44	-17	-17	79	79	
ORP (post)	mV	197	100%				3	3												
ORP (pre)	mV	193	100%				3	3												
pH	Std units	8.37	100%				90	90	6.98	6.98	6.79	7.2	7.2		7.57	7.74	7.74	7.1	7.1	
pH (post)	Std units	7.38	100%				3	3												
pH (pre)	Std units	7.38	100%				3	3												
Sulfide	MG/L	1.04	89%				72	81			0.2	0.15	0.15		0	0.03	0.03	0.04	0.04	
Temperature	DEG C	26.55	100%				93	93	5.08	5.08	5.3	8.4	8.4		3.3	5.1	5.1	4.3	4.3	
Turbidity	NTU	195	100%				90	90	0.6	0.6	5.38	3.11	3.11		1.48	3.67	3.67	2.2	2.2	
Turbidity (post)	NTU	7.6	100%				2	2												
Turbidity (pre)	NTU	5.7	100%				1	1												

Notes:
1. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
2. Shading indicates concentration above cleanup goal.

U = compound was not detected
J = the reported value is an estimated concentration
UJ = the compound was not detected; the associated reporting limit is approximate
ND = Non-detect

SA = Sample
DU = Duplicate

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-25 MW25-3 GROUNDWATER 25LM20011 0-0.1 8/11/2006 SA LTM 2 Total Value	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20036 0-0.1 3/4/2008 SA LTM 4 Total Value	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20046 0-0.1 4/29/2009 SA LTM 5 Total Value	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20060 0-0.1 1/12/2010 SA LTM 6 Total Value	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20068 0-0 8/4/2010 SA LTM 7 Total Value	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20075 8.8-8.8 2/8/2011 SA LTM 8 Total Value	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20086 8.79-8.79 2/29/2012 SA LTM 9 Total Value	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20087 8.79-8.79 2/29/2012 DU LTM 9 Total Value	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20097 8.8-8.8 5/9/2013 SA LTM 10 Total Value	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20110 8.8-8.8 6/18/2014 SA LTM 11 Total Value	Qual	SEAD-25 MW25-3 GROUNDWATER 25LM20114 8.8-8.8 3/18/2015 SA LTM 12 Total Value	Qual	GR
Volatile Organic Compounds																																				
1,1,1-Trichloroethane	UG/L	0.62	2%	GA	5	0	2	107	1	U	1	U	1	U	0.32	U	1.6	U	0.2	UJ	0.2	U	0.2	U	0.5	U	0.5	U	0.37	U	
1,1,2,2-Tetrachloroethane	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.09	U	0.45	U	0.38	UJ	0.38	U	0.38	U	0.18	U	0.18	U	0.62	U	
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/L	0	0%	GA	5	0	0	102	1	U	1	U	1	U	0.4	U	2	U	0.31	U	0.31	UJ	0.31	UJ	0.5	U	0.5	U	0.36	U	
1,1,2-Trichloroethane	UG/L	0	0%	GA	1	0	0	107	1	U	1	U	1	U	0.2	U	1	U	0.33	UJ	0.33	U	0.33	U	0.13	U	0.13	U	0.33	U	
1,1-Dichloroethane	UG/L	3.5	9%	GA	5	0	10	107	1	U	1	U	1	U	0.14	U	0.71	U	0.21	UJ	0.21	U	0.21	U	0.25	U	0.25	U	0.38	U	
1,1-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.37	U	1.9	U	0.35	UJ	0.35	U	0.35	U	0.11	U	0.11	U	0.36	U	
1,2,4-Trichlorobenzene	UG/L	0	0%	GA	5	0	0	102	1	U	1	U	1	U	0.19	U	0.95	U	0.37	UJ	0.37	U	0.37	U	0.25	UJ	0.25	UJ	2.5	U	
1,2,4-Trimethylbenzene	UG/L	0.45	7%	GA	5	0	2	30	1	U									0.19	UJ	0.19	U	0.19	U							
1,2-Dibromo-3-chloropropane	UG/L	0	0%	GA	0.04	0	0	102	1	U	2	U	2	U	0.43	U	2.2	U	0.5	U	0.5	U	0.5	U	0.44	U	0.44	U	1.1	U	
1,2-Dibromoethane	UG/L	0	0%	GA	0.0006	0	0	107	1	U	1	U	1	U	0.18	U	0.9	U	0.22	UJ	0.22	U	0.22	U	0.25	U	0.25	U	0.44	U	
1,2-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	1	U	1	U	0.4	U	2	U	0.15	UJ	0.15	U	0.15	U	0.21	U	0.21	U	0.37	U	
1,2-Dichloroethane	UG/L	0.49	3%	GA	0.6	0	3	107	1	U	1	U	1	U	0.14	U	0.71	U	0.2	UJ	0.2	U	0.2	U	0.1	U	0.1	U	0.5	U	
1,2-Dichloroethene (total)	UG/L	15	15%	GA	5	2	3	20											0.21	UJ	0.21	UJ	0.21	UJ							
1,2-Dichloropropane	UG/L	0	0%	GA	1	0	0	107	1	U	1	U	1	U	0.15	U	0.75	U	0.25	UJ	0.25	U	0.25	U	0.13	U	0.13	U	0.67	U	
1,3,5-Trimethylbenzene	UG/L	0	0%	GA	5	0	0	30	1	U									0.2	UJ	0.2	U	0.2	U							
1,3-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	1	U	1	U	0.36	U	1.8	U	0.26	UJ	0.26	U	0.26	U	0.25	U	0.25	U	0.43	U	
1,4-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	1	U	1	U	0.34	U	1.8	U	0.24	UJ	0.24	U	0.24	U	0.28	U	0.28	U	0.46	U	
Acetone	UG/L	11	7%				7	107	5.9	UJ	10	UJ	5	U	5	U	8	U	2.2	U	2.2	U	2.2	U	5	U	5	U	7	U	
Benzene	UG/L	62	29%	GA	1	19	31	107	1	U	1	U	**1.7**		0.18	U	0.9	U	0.26	UJ	0.68	J	0.68	J	0.82	J	**1.8**		0.43	U	
Bromodichloromethane	UG/L	0	0%	MCL	80	0	0	107	1	U	1	U	1	U	0.17	U	0.86	U	0.33	UJ	0.33	U	0.33	U	0.25	U	0.25	U	0.44	U	
Bromoform	UG/L	0	0%	MCL	80	0	0	107	1	U	1	UJ	1	U	0.2	U	1	U	0.23	UJ	0.23	U	0.23	U	0.5	UJ	0.5	U	0.43	U	
Carbon disulfide	UG/L	0.61	2%				2	107	1	U	1	U	1	U	0.36	U	1.8	U	0.25	U	0.25	U	0.25	U	0.6	U	0.6	U	1	U	
Carbon tetrachloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.36	U	1.8	U	0.22	UJ	0.22	U	0.22	U	0.5	UJ	0.5	U	0.33	U	
Chlorobenzene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.26	U	1.3	U	0.22	UJ	0.22	U	0.22	U	0.25	U	0.25	U	0.26	U	
Chlorodibromomethane	UG/L	0	0%	MCL	80	0	0	107	1	U	1	U	1	U	0.11	U	0.55	U	0.3	UJ	0.3	U	0.3	U	0.1	UJ	0.1	U	0.32	U	
Chloroethane	UG/L	0.67	2%	GA	5	0	2	107	1	U	2	U	1	U	0.21	U	1.1	U	0.55	U	0.55	U	0.55	U	2	UJ	2	U	2.5	U	
Chloroform	UG/L	0.32	1%	GA	7	0	1	107	1	U	1	U	1	U	0.16	U	0.8	U	0.32	UJ	0.32	U	0.32	U	0.14	U	0.14	U	0.5	U	
Cis-1,2-Dichloroethene	UG/L	19	14%	GA	5	4	15	107	1	U	1	U	1	U	0.14	U	0.71	U	0.21	UJ	0.21	U	0.21	U	0.15	U	0.15	U	0.41	U	
Cis-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	1	U	1	U	1	U	0.14	U	0.71	U	0.19	UJ	0.19	UJ	0.19	UJ	0.11	U	0.11	U	0.4	U	
Cyclohexane	UG/L	8.6	11%				11	102	1	U	1	U	1	U	0.14	U	0.71	U	0.31	UJ	0.31	U	0.31	U	0.25	U	0.25	U	0.39	U	
Dichlorodifluoromethane	UG/L	0	0%	GA	5	0	0	82	1	U	1	UJ	1	U	0.18	U	0.9	U							0.25	U	0.25	U	0.6	U	
Diisoproply Ether	UG/L	0	0%				0	20											0.21	U	0.21	U	0.21	U							
Ethyl benzene	UG/L	26	16%	GA	5	12	17	107	1	U	1	U	1	U	0.42	U	2.1	U	0.21	UJ	0.21	U	0.21	U	0.11	U	1		0.33	U	
Isopropylbenzene	UG/L	2.6	9%	GA	5	0	10	107	1	U	1	U	1	U	0.34	U	1.8	U	0.23	UJ	0.23	U	0.23	U	0.1	U	0.39	J	0.35	U	
Meta/Para Xylene	UG/L	19	11%	GA	5	4	7	61			1	U	2	U	0.81	U	4.1	U	0.59	UJ	0.59	U	0.59	U							
Methyl Acetate	UG/L	0	0%				0	102	1	U	10	U	2	U	0.48	U	2.4	U	0.53	U	0.53	U	0.53	U	0.19	UJ	0.19	U	1.8	U	
Methyl bromide	UG/L	0	0%	GA	5	0	0	107	1	UJ	2	U	1	UJ	0.4	U	2	U	0.49	U	0.49	UJ	0.49	UJ	2	U	2	U	2.5	U	
Methyl butyl ketone	UG/L	1.9	1%				1	107	5	U	5	U	5	U	0.4	U	2	U	1.7	U	1.7	U	1.7	U	1	U	1	U	2	U	
Methyl chloride	UG/L	0	0%	GA	5	0	0	107	1	U	2	U	1	U	0.18	U	0.9	U	0.36	U	0.36	U	0.36	U	0.33	U	0.33	U	0.4	U	
Methyl cyclohexane	UG/L	4.2	7%				7	102	1	U	1	U	1	U	0.16	U	0.8	U	0.3	U	0.3	U	0.3	U	0.1	U	0.1	U	0.43	U	
Methyl ethyl ketone	UG/L	9	10%				11	107	5	U	5	U	5	U	1	U	5	U	1.3	U	1.3	U	1.3	U	1	U	1	U	3.4	U	
Methyl isobutyl ketone	UG/L	0	0%				0	107	5	U	5	U	5	U	0.34	U	1.8	U	1.3	U	1.3	U	1.3	U	1	U	1	U	2.1	U	
Methyl Tertbutyl Ether	UG/L	0	0%				0	107	1	U	1	U	1	U	0.13	U	0.65	U	0.36	U	0.36	U	0.36	U	0.2	U	0.2	U	0.3	U	
Methylene chloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.13	U	0.65	U	1.1	U	1.1	U	1.1	U	1	U	1	U	2.5	U	
Naphthalene	UG/L	0.23	4%				1	25											0.3	UJ	0.3	U	0.3	U							
n-Butylbenzene	UG/L	0	0%	GA	5	0	0	20											0.23	UJ	0.23	U	0.23	U							
Ortho Xylene	UG/L	6.4	8%	GA	5	3	5	61			1	U	1	U	0.4	U	2	U	0.25	UJ	0.25	U	0.25	U							
p-Isopropyltoluene	UG/L	0	0%	GA	5	0	0	10	1	U																					
Propylbenzene	UG/L	0	0%	GA	5	0	0	10	1	U																					
sec-Butylbenzene	UG/L	0	0%	GA	5	0	0	20											0.21	UJ	0.21	U	0.21	U							
Styrene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.36	U	1.8	U	0.23	UJ	0.23	U	0.23	U	0.11	U	0.11	U	0.27	U	
tert-Butylbenzene	UG/L	0	0%	GA	5	0	0	20											0.31	UJ	0.31	U	0.31	U							
Tetrachloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.42	U	2.1	U	0.4	U	0.4	U	0.4	U	0.15	U	0.15	U	0.74	U	
Toluene	UG/L	14	11%	GA	5	5	12	107	1	U	1	U	1	U	0.21	U	1.1	U	0.27	UJ	0.27	U	0.27	U	0.33	U	0.4	J	0.48	U	
Total Xylenes	UG/L	62	11%	GA	5	3	7	66	3	U									0.25	UJ	0.25	U	0.25	U	0.2	U	2.3		0.23	U	
Trans-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	1	U	0.16	U	0.8	U	0.25	U	0.25	U	0.25	U	0.2	U	0.2	U	0.37	U	
Trans-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	1	U	1	U	1	U	0.17	U	0.86	U	0.2	UJ	0.2	UJ	0.2	UJ	0.21	U	0.21	U	0.42	U	
Trichloroethene	UG/L	2	10%	GA	5	0	11	107	1	U	1	U	1	U	0.19	U	0.95	U	0.28	UJ	0.28	U	0.28	U	0.13	U	0.13	U	0.48	U	
Trichlorofluoromethane	UG/L	0	0%	GA	5	0	0	102	1	U	1	U	1	U	0.16	U	0.8	U	0.24	U	0.24	U	0.24	U	0.25	U	0.25	U	0.42	U	
Vinyl chloride	UG/L	2.6	6%	GA	2	2	6	107	1	U	1	U	1	U	0.22	U	1.1	U	0.25	U	0.25	U	0.25	U	0.18	U	0.18	U	0.5	U	

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-3		MW25-3		MW25-3		MW25-3		MW25-3		MW25-3		MW25-3		MW25-3		MW25-3		MW25-3	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20011		25LM20036		25LM20046		25LM20060		25LM20068		25LM20075		25LM20086		25LM20087		25LM20097		25LM20110		25LM20114	
Sample Depth Interval (FT)									0-0.1		0-0.1		0-0.1		0-0.1		0-0		8.8-8.8		8.79-8.79		8.79-8.79		8.8-8.8		8.8-8.8		8.8-8.8	
Sample Date									8/11/2006		3/4/2008		4/29/2009		1/12/2010		8/4/2010		2/8/2011		2/29/2012		2/29/2012		5/9/2013		6/18/2014		3/18/2015	
QC Type									SA		SA		SA		SA		SA		SA		SA		DU		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									2		4		5		6		7		8		9		9		10		11		12	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Semivolatile Organic Compounds																														
1,1'-Biphenyl	UG/L	0	0%	GA	5	0	0	18	10	U																				
2,4,5-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18	10	U																				
2,4,6-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18	10	U																				
2,4-Dichlorophenol	UG/L	0	0%	GA	5	0	0	18	10	U																				
2,4-Dimethylphenol	UG/L	0	0%				0	18	10	U																				
2,4-Dinitrophenol	UG/L	0	0%				0	18	48	U																				
2,4-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18	10	U																				
2,6-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18	10	U																				
2-Chloronaphthalene	UG/L	0	0%				0	18	10	U																				
2-Chlorophenol	UG/L	0	0%				0	18	10	U																				
2-Methylnaphthalene	UG/L	0	0%				0	18	10	U																				
2-Methylphenol	UG/L	0	0%				0	18	10	U																				
2-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	48	U																				
2-Nitrophenol	UG/L	0	0%	GA	1	0	0	18	10	U																				
3,3'-Dichlorobenzidine	UG/L	0	0%	GA	5	0	0	18	19	U																				
3-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	48	U																				
4,6-Dinitro-2-methylphenol	UG/L	0	0%	GA	1	0	0	18	48	U																				
4-Bromophenyl phenyl ether	UG/L	0	0%				0	18	10	U																				
4-Chloro-3-methylphenol	UG/L	0	0%	GA	1	0	0	18	10	U																				
4-Chloroaniline	UG/L	0	0%	GA	5	0	0	18	10	U																				
4-Chlorophenyl phenyl ether	UG/L	0	0%				0	18	10	U																				
4-Methylphenol	UG/L	0	0%				0	18	10	U																				
4-Nitroaniline	UG/L	0	0%	GA	5	0	0	18	48	U																				
4-Nitrophenol	UG/L	0	0%	GA	1	0	0	18	48	U																				
Acenaphthene	UG/L	0.5	6%				1	18	10	U																				
Acenaphthylene	UG/L	2	22%				4	18	10	U																				
Acetophenone	UG/L	0	0%				0	18	10	U																				
Anthracene	UG/L	1	6%				1	18	10	U																				
Atrazine	UG/L	0	0%	GA	7.5	0	0	18	10	U																				
Benzaldehyde	UG/L	0	0%				0	18	48	U																				
Benzo(a)anthracene	UG/L	0	0%				0	18	10	U																				
Benzo(a)pyrene	UG/L	0	0%	GA	0		0	18	10	U																				
Benzo(b)fluoranthene	UG/L	0	0%				0	18	10	U																				
Benzo(ghi)perylene	UG/L	0.6	6%				1	18	10	U																				
Benzo(k)fluoranthene	UG/L	0	0%				0	18	10	U																				
Bis(2-Chloroethoxy)methane	UG/L	0	0%	GA	5	0	0	18	10	U																				
Bis(2-Chloroethyl)ether	UG/L	0	0%	GA	1	0	0	18	10	U																				
Bis(2-Chloroisopropyl)ether	UG/L	0	0%	GA	5	0	0	18	10	U																				
Bis(2-Ethylhexyl)phthalate	UG/L	11	6%	GA	5	1	1	18	10	U																				
Butylbenzylphthalate	UG/L	2	6%				1	18	10	U																				
Caprolactam	UG/L	0	0%				0	18	10	U																				
Carbazole	UG/L	0	0%				0	18	10	U																				
Chrysene	UG/L	0	0%				0	18	10	U																				
Dibenz(a,h)anthracene	UG/L	0	0%				0	18	10	U																				
Dibenzofuran	UG/L	0	0%				0	18	10	U																				
Diethyl phthalate	UG/L	0	0%				0	18	10	U																				
Dimethylphthalate	UG/L	0	0%				0	18	10	U																				
Di-n-butylphthalate	UG/L	0	0%	GA	50	0	0	18	10	U																				
Di-n-octylphthalate	UG/L	0	0%				0	18	10	U																				
Fluoranthene	UG/L	0	0%				0	18	10	U																				
Fluorene	UG/L	0	0%				0	18	10	U																				
Hexachlorobenzene	UG/L	0	0%	GA	0.04	0	0	18	10	U																				
Hexachlorobutadiene	UG/L	0	0%	GA	0.5	0	0	18	10	U																				
Hexachlorocyclopentadiene	UG/L	0	0%	GA	5	0	0	18	43	U																				
Hexachloroethane	UG/L	0	0%	GA	5	0	0	18	10	U																				
Indeno(1,2,3-cd)pyrene	UG/L	0	0%				0	18	10	U																				
Isophorone	UG/L	0	0%				0	18	10	U																				
Naphthalene	UG/L	2	6%				1	18	10	U																				
Nitrobenzene	UG/L	0	0%	GA	0.4	0	0	18	10	U																				
N-Nitroso-di-n-propylamine	UG/L	0	0%				0	18	10	U																				
N-Nitrosodiphenylamine	UG/L	0	0%				0	18	10	U																				
Pentachlorophenol	UG/L	0	0%	GA	1	0	0	18	48	U																				
Phenanthrene	UG/L	0	0%				0	18	10	U																				
Phenol	UG/L	0	0%	GA	1	0	0	18	10	U																				
Pyrene	UG/L	0	0%				0	18	10	U																				

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-3		MW25-3		MW25-3		MW25-3		MW25-3		MW25-3		MW25-3		MW25-3		MW25-3		MW25-3	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20011		25LM20036		25LM20046		25LM20060		25LM20068		25LM20075		25LM20086		25LM20087		25LM20097		25LM20110		25LM20114	
Sample Depth Interval (FT)									0-0.1		0-0.1		0-0.1		0-0.1		0-0		8.8-8.8		8.79-8.79		8.79-8.79		8.8-8.8		8.8-8.8		8.8-8.8	
Sample Date									8/11/2006		3/4/2008		4/29/2009		1/12/2010		8/4/2010		2/8/2011		2/29/2012		2/29/2012		5/9/2013		6/18/2014		3/18/2015	
QC Type									SA		SA		SA		SA		SA		SA		SA		DU		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									2		4		5		6		7		8		9		9		10		11		12	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																														
Iron	UG/L	15,700	85%	GA	300	56	84	99	**3,820**		107		**1,570**		**702**				**463**		**458**	**J**	**530**	**J**	**2,200**				50	U
Sodium	UG/L	58,100	100%	GA	20,000	14	99	99	11,300	J	5,540		9,000		7,370				7,990		5,980		5,960		8,900				6,000	
Wet Chemistry - MEE																														
Chloride	MG/L	97.9	78%	GA	250	0	78	100	1.5	J	2.66		3.3		2.8				3.2		1.5	J	1.4	J	1	U			1.1	
Ethane	UG/L	1.1	5%				5	104	2	U	1	U	1	U	0.16	U	0.16	U	0.58	U	0.58	U	0.58	U	0.55	U	0.55	U	0.55	U
Ethene	UG/L	4.6	5%				5	104	2	U	1	U	1	U	0.17	U	0.17	U	0.69	U	0.69	U	0.69	U	0.5	U	0.5	U	0.5	U
Methane	UG/L	170	57%				59	104	2	U	0.34	J	13		0.14	U	12		1.5	J	18	J	18	J	0.29	U	11		1.7	
Nitrate	MG/L	6.4	68%	GA	10	0	45	66					0.05	U	0.05	UJ			0.057		0.0152	U	0.0152	U	0.019	J			0.69	
Nitrate Nitrogen	MG/L	1	45%				13	29	0.05	U	0.098	J																		
Nitrate/Nitrite Nitrogen	MG/L	1	64%	GA	10	0	16	25			0.098				0.003	UJ														
Nitrite	MG/L	0.73	27%	GA	1	0	18	66					0.01	U	0.007	UJ			0.00321	U	0.022	J	0.023	J	0.01	U			0.01	U
Nitrite Nitrogen	MG/L	0.087	3%				1	29	0.05	U	0.01	UJ																		
Sulfate	MG/L	182	100%	GA	250	0	100	100	44.9		100		122		182	J			110	J	50	J	50	J	100				70	
Field Measurement - Hach Kit																														
Conductivity	S/m	1.26	100%				90	90	0.686		0.675		0.627		0.741		1.26		0.851		0.766		0.766		0.808				0.686	
Conductivity (post)	S/m	0.844	100%				3	3																						
Conductivity (pre)	S/m	0.83	100%				3	3																						
Dissolved Oxygen	MG/L	12.6	100%				86	86	3.6		0.87		0.19		1.78		0		0.37		0.1		0.1		0.25				4.06	
Dissolved Oxygen (post)	MG/L	5.17	100%				3	3																						
Dissolved Oxygen (pre)	MG/L	5.36	100%				3	3																						
Nitrate Nitrogen	MG/L	0.5	100%				3	3																						
Nitrite Nitrogen	MG/L	0.5	100%				3	3																						
ORP	mV	259	100%				90	90	77.9		124		-102		-63		-124		-85		-141		-141		-79				189	
ORP (post)	mV	197	100%				3	3																						
ORP (pre)	mV	193	100%				3	3																						
pH	Std units	8.37	100%				90	90	7.02		7.15		7.03		6.51		6.84		6.99		6.94		6.94		6.99				7.29	
pH (post)	Std units	7.38	100%				3	3																						
pH (pre)	Std units	7.38	100%				3	3																						
Sulfide	MG/L	1.04	89%				72	81	0.03		0.01		0.42		0.04						0.46		0.46		0.03				0	
Temperature	DEG C	26.55	100%				93	93	21.54		3.5		7.9		4.9		20.6		4.5		4.6		4.6		7.8				3.1	
Turbidity	NTU	195	100%				90	90	1.2		2		0.35		3		2.37		3.31		1.99		1.99		1.5				1.79	
Turbidity (post)	NTU	7.6	100%				2	2																						
Turbidity (pre)	NTU	5.7	100%				1	1																						

Notes:
1. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
2. Shading indicates concentration above cleanup goal.

U = compound was not detected
J = the reported value is an estimated concentration
UJ = the compound was not detected; the associated reporting limit is approximate
ND = Non-detect

SA = Sample
DU = Duplicate

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Area	Loc ID	Matrix	Sample ID	Sample Depth Interval (FT)	Sample Date	QC Type	Study ID	Sample Round	Filtered	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
									SEAD-25	MW25-3	GROUNDWATER	25LM20115	8.8-8.8	3/18/2015	DU	LTM	12	Total																	
									SEAD-25	MW25-3	GROUNDWATER	25LM20121	5.23-5.23	3/17/2016	SA	LTM	13	Total																	
									SEAD-25	MW25-8	GROUNDWATER	25LM20003	0-0.1	1/31/2006	SA	LTM	1	Total																	
									SEAD-25	MW25-8	GROUNDWATER	25LM20012	0-0.1	8/11/2006	SA	LTM	2	Total																	
									SEAD-25	MW25-8	GROUNDWATER	25LM20037	0-0.1	3/4/2008	SA	LTM	4	Total																	
									SEAD-25	MW25-8	GROUNDWATER	25LM20047	0-0.1	4/29/2009	SA	LTM	5	Total																	
									SEAD-25	MW25-8	GROUNDWATER	25LM20059	0-0.1	1/13/2010	SA	LTM	6	Total																	
									SEAD-25	MW25-8	GROUNDWATER	25LM20092	4.41-4.41	2/29/2012	SA	LTM	9	Total																	
									SEAD-25	MW25-8	GROUNDWATER	25LM20103	4.25-4.25	5/7/2013	SA	LTM	10	Total																	
									SEAD-25	MW25-9	GROUNDWATER	25LM20004	0-0.1	1/31/2006	SA	LTM	1	Total																	
									SEAD-25	MW25-9	GROUNDWATER	25LM20013	0-0.1	8/9/2006	SA	LTM	2	Total																	
Volatile Organic Compounds																																		
1,1,1-Trichloroethane	UG/L	0.62	2%	GA	5	0	2	107	0.37	U	0.37	U	1	U	1	U	1	U	1	U	0.32	U	0.2	U	0.5	UJ	0.62	J	1	U
1,1,2,2-Tetrachloroethane	UG/L	0	0%	GA	5	0	0	107	0.62	U	0.62	U	1	U	1	U	1	U	1	U	0.09	U	0.38	U	0.18	U	1	U	1	U
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/L	0	0%	GA	5	0	0	102	0.36	U	0.36	U	1	U	1	U	1	U	1	U	0.4	U	0.31	UJ	0.5	U	1	U	1	U
1,1,2-Trichloroethane	UG/L	0	0%	GA	1	0	0	107	0.33	U	0.33	U	1	U	1	U	1	U	1	U	0.2	U	0.33	U	0.13	U	1	U	1	U
1,1-Dichloroethane	UG/L	3.5	9%	GA	5	0	10	107	0.38	U	0.38	U	1	U	1	U	1	U	1	U	0.14	U	0.21	U	0.25	U	1		1	U
1,1-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	0.36	U	0.36	U	1	U	1	U	1	U	1	U	0.37	U	0.35	U	0.11	U	1	U	1	U
1,2,4-Trichlorobenzene	UG/L	0	0%	GA	5	0	0	102	2.5	U	2.5	U	1	U	1	U	1	U	1	U	0.19	U	0.37	U	0.25	U	1	U	1	U
1,2,4-Trimethylbenzene	UG/L	0.45	7%	GA	5	0	2	30									1	U					0.19	U					1	U
1,2-Dibromo-3-chloropropane	UG/L	0	0%	GA	0.04	0	0	102	1.1	U	1.1	U	1	U	1	U	2	U	2	U	0.43	U	0.5	U	0.44	U	1	U	1	U
1,2-Dibromoethane	UG/L	0	0%	GA	0.0006	0	0	107	0.44	U	0.44	U	1	U	1	U	1	U	1	U	0.18	U	0.22	U	0.25	U	1	U	1	U
1,2-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.37	U	0.37	U	1	U	1	U	1	U	1	U	0.4	U	0.15	U	0.21	U	1	U	1	U
1,2-Dichloroethane	UG/L	0.49	3%	GA	0.6	0	3	107	0.5	U	0.5	U	1	U	1	U	1	U	1	U	0.14	U	0.2	U	0.1	U	0.49	J	1	U
1,2-Dichloroethene (total)	UG/L	15	15%	GA	5	2	3	20															0.21	UJ						
1,2-Dichloropropane	UG/L	0	0%	GA	1	0	0	107	0.67	U	0.67	U	1	U	1	U	1	U	1	U	0.15	U	0.25	U	0.13	U	1	U	1	U
1,3,5-Trimethylbenzene	UG/L	0	0%	GA	5	0	0	30									1	U					0.2	U					1	U
1,3-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.43	U	0.43	U	1	U	1	U	1	U	1	U	0.36	U	0.26	U	0.25	U	1	U	1	U
1,4-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	0.46	U	0.46	U	1	U	1	U	1	U	1	U	0.34	U	0.24	U	0.28	U	1	U	1	U
Acetone	UG/L	11	7%				7	107	7	U	7	U	5	U	5	U	10	UJ	10	U	5	U	2.2	U	5	U	5	U	63	UJ
Benzene	UG/L	62	29%	GA	1	19	31	107	0.43	U	0.43	U	1	U	1	U	1	U	1	U	0.18	U	0.26	U	0.25	U	**33**		0.58	J
Bromodichloromethane	UG/L	0	0%	MCL	80	0	0	107	0.44	U	0.44	U	1	U	1	U	1	U	1	U	0.17	U	0.33	U	0.25	UJ	1	U	1	U
Bromoform	UG/L	0	0%	MCL	80	0	0	107	0.43	U	0.43	UJ	1	U	1	U	1	UJ	1	U	0.2	U	0.23	U	0.5	U	1	U	1	U
Carbon disulfide	UG/L	0.61	2%				2	107	1	U	1	U	1	U	1	U	1	U	1	U	0.36	U	0.25	U	0.6	U	1	U	1	U
Carbon tetrachloride	UG/L	0	0%	GA	5	0	0	107	0.33	U	0.33	U	1	U	1	U	1	U	1	U	0.36	U	0.22	U	0.5	UJ	1	U	1	U
Chlorobenzene	UG/L	0	0%	GA	5	0	0	107	0.26	U	0.26	U	1	U	1	U	1	U	1	U	0.26	U	0.22	U	0.25	U	1	U	1	U
Chlorodibromomethane	UG/L	0	0%	MCL	80	0	0	107	0.32	U	0.32	U	1	U	1	U	1	U	1	U	0.11	U	0.3	U	0.1	U	1	U	1	U
Chloroethane	UG/L	0.67	2%	GA	5	0	2	107	2.5	U	2.5	U	1	U	1	U	2	U	1	U	0.21	U	0.55	U	2	U	1	U	1	UJ
Chloroform	UG/L	0.32	1%	GA	7	0	1	107	0.5	U	0.5	U	1	U	1	U	1	U	1	U	0.16	U	0.32	U	0.14	U	1	U	1	U
Cis-1,2-Dichloroethene	UG/L	19	14%	GA	5	4	15	107	0.41	U	0.41	U	1	U	1	U	1	U	1	U	0.14	U	0.21	U	0.15	U	2.8		1	U
Cis-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	0.4	U	0.4	U	1	U	1	U	1	U	1	U	0.14	U	0.19	UJ	0.11	U	1	U	1	U
Cyclohexane	UG/L	8.6	11%				11	102	0.39	U	0.39	U	1	U	1	U	1	U	1	U	0.14	U	0.31	U	0.25	U	8	J	1	U
Dichlorodifluoromethane	UG/L	0	0%	GA	5	0	0	82	0.6	U	0.6	U	1	UJ	1	U	1	UJ	1	U	0.18	U			0.25	U	1	UJ	1	U
Diisoproply Ether	UG/L	0	0%				0	20															0.21	U						
Ethyl benzene	UG/L	26	16%	GA	5	12	17	107	0.33	U	0.33	U	1	U	1	U	1	U	1	U	0.42	U	0.21	U	0.11	U	**15**		1	U
Isopropylbenzene	UG/L	2.6	9%	GA	5	0	10	107	0.35	U	0.35	U	1	U	1	U	1	U	1	U	0.34	U	0.23	U	0.1	U	2.6		1	U
Meta/Para Xylene	UG/L	19	11%	GA	5	4	7	61									1	U	2	U	0.81	U	0.59	U						
Methyl Acetate	UG/L	0	0%				0	102	1.8	U	1.8	U	1	U	1	U	10	U	2	U	0.48	U	0.53	U	0.19	U	1	U	1	U
Methyl bromide	UG/L	0	0%	GA	5	0	0	107	2.5	U	2.5	UJ	1	U	1	U	1	UJ	1	UJ	0.4	U	0.49	UJ	2	U	1	U	1	U
Methyl butyl ketone	UG/L	1.9	1%				1	107	2	U	2	U	5	U	5	U	5	U	5	U	0.4	U	1.7	U	1	U	5	U	5	U
Methyl chloride	UG/L	0	0%	GA	5	0	0	107	0.4	U	0.4	U	1	U	1	U	2	U	1	U	0.18	U	0.36	U	0.33	U	1	U	1	U
Methyl cyclohexane	UG/L	4.2	7%				7	102	0.43	U	0.43	U	1	U	1	U	1	U	1	U	0.16	U	0.3	U	0.1	U	1.9	J	1	U
Methyl ethyl ketone	UG/L	9	10%				11	107	3.4	U	3.4	U	5	U	5	U	5	U	2.3	J	1	U	1.3	U	1	U	5	U	5	UJ
Methyl isobutyl ketone	UG/L	0	0%				0	107	2.1	U	2.1	U	5	U	5	U	5	U	5	U	0.34	U	1.3	U	1	U	5	U	5	U
Methyl Tertbutyl Ether	UG/L	0	0%				0	107	0.3	U	0.3	U	1	U	1	U	1	U	1	U	0.13	U	0.36	U	0.2	U	1	U	1	U
Methylene chloride	UG/L	0	0%	GA	5	0	0	107	2.5	U	2.5	U	1	U	1	U	1	U	1	U	0.13	U	1.1	U	1	U	1	U	1	UJ
Naphthalene	UG/L	0.23	4%				1	25															0.3	U						
n-Butylbenzene	UG/L	0	0%	GA	5	0	0	20															0.23	U						
Ortho Xylene	UG/L	6.4	8%	GA	5	3	5	61									1	U	1	U	0.4	U	0.25	U						
p-Isopropyltoluene	UG/L	0	0%	GA	5	0	0	10									1	U											1	U
Propylbenzene	UG/L	0	0%	GA	5	0	0	10									1	U											1	U
sec-Butylbenzene	UG/L	0	0%	GA	5	0	0	20															0.21	U						
Styrene	UG/L	0	0%	GA	5	0	0	107	0.27	U	0.27	U	1	U	1	U	1	U	1	U	0.36	U	0.23	U	0.11	U	1	U	1	U
tert-Butylbenzene	UG/L	0	0%	GA	5	0	0	20															0.31	U						
Tetrachloroethene	UG/L	0	0%	GA	5	0	0	107	0.74	U	0.74	UJ	1	U	1	U	1	U	1	U	0.42	U	0.4	U	0.15	U	1	U	1	U
Toluene	UG/L	14	11%	GA	5	5	12	107	0.48	U	0.48	U	1	U	1	U	1	U	1	U	0.21	U	0.27	U	0.33	U	**14**		1	U
Total Xylenes	UG/L	62	11%	GA	5	3	7	66	0.23	U	0.23	U	3	U	3	U							0.25	U	0.2	U	**62**		3	U
Trans-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	0.37	U	0.37	U	1	U	1	U	1	U	1	U	0.16	U	0.25	U	0.2	U	1	U	1	U
Trans-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	0.42	U	0.42	U	1	U	1	U	1	U	1	U	0.17	U	0.2	UJ	0.21	UJ	1	U	1	U
Trichloroethene	UG/L	2	10%	GA	5	0	11	107	0.48	U	0.48	U	1	U	1	U	1	U	1	U	0.19	U	0.28	U	0.13	U	0.53	J	1	U
Trichlorofluoromethane	UG/L	0	0%	GA	5	0	0	102	0.42	U	0.42	U	1	UJ	1	U	1	U	1	U	0.16	U	0.24	U	0.25	U	1	UJ	1	U
Vinyl chloride	UG/L	2.6	6%	GA	2	2	6	107	0.5	U	0.5	U	1	U	1	U	1	U	1	U	0.22	U	0.25	U	0.18	U	1	U	1	U

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-3		MW25-3		MW25-8		MW25-8		MW25-8		MW25-8		MW25-8		MW25-8		MW25-9		MW25-9	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	GR
Sample ID									25LM20115		25LM20121		25LM20003		25LM20012		25LM20037		25LM20047		25LM20059		25LM20092		25LM20103		25LM20004		25LM20013	
Sample Depth Interval (FT)									8.8-8.8		5.23-5.23		0-0.1		0-0.1		0-0.1		0-0.1		0-0.1		4.41-4.41		4.25-4.25		0-0.1		0-0.1	
Sample Date									3/18/2015		3/17/2016		1/31/2006		8/11/2006		3/4/2008		4/29/2009		1/13/2010		2/29/2012		5/7/2013		1/31/2006		8/9/2006	
QC Type									DU		SA		SA		SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									12		13		1		2		4		5		6		9		10		1		2	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Semivolatile Organic Compounds																														
1,1'-Biphenyl	UG/L	0	0%	GA	5	0	0	18					9	U	10	U											10	U	10	U
2,4,5-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18					9	U	10	U											10	U	10	U
2,4,6-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18					9	U	10	U											10	U	10	U
2,4-Dichlorophenol	UG/L	0	0%	GA	5	0	0	18					9	U	10	U											10	U	10	U
2,4-Dimethylphenol	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
2,4-Dinitrophenol	UG/L	0	0%				0	18					47	U	49	U											48	U	48	U
2,4-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18					9	U	10	U											10	U	10	U
2,6-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18					9	U	10	U											10	U	10	U
2-Chloronaphthalene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
2-Chlorophenol	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
2-Methylnaphthalene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
2-Methylphenol	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
2-Nitroaniline	UG/L	0	0%	GA	5	0	0	18					47	U	49	U											48	U	48	U
2-Nitrophenol	UG/L	0	0%	GA	1	0	0	18					9	U	10	U											10	U	10	U
3,3'-Dichlorobenzidine	UG/L	0	0%	GA	5	0	0	18					19	U	20	U											19	U	19	U
3-Nitroaniline	UG/L	0	0%	GA	5	0	0	18					47	U	49	U											48	U	48	U
4,6-Dinitro-2-methylphenol	UG/L	0	0%	GA	1	0	0	18					47	U	49	U											48	U	48	U
4-Bromophenyl phenyl ether	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
4-Chloro-3-methylphenol	UG/L	0	0%	GA	1	0	0	18					9	U	10	U											10	U	10	U
4-Chloroaniline	UG/L	0	0%	GA	5	0	0	18					9	U	10	U											10	U	10	U
4-Chlorophenyl phenyl ether	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
4-Methylphenol	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
4-Nitroaniline	UG/L	0	0%	GA	5	0	0	18					47	U	49	U											48	U	48	U
4-Nitrophenol	UG/L	0	0%	GA	1	0	0	18					47	U	49	U											48	U	48	U
Acenaphthene	UG/L	0.5	6%				1	18					0.5	J	10	U											10	U	10	U
Acenaphthylene	UG/L	2	22%				4	18					2	J	10	U											1	J	10	U
Acetophenone	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Anthracene	UG/L	1	6%				1	18					1	J	10	U											10	U	10	U
Atrazine	UG/L	0	0%	GA	7.5	0	0	18					9	U	10	U											10	U	10	U
Benzaldehyde	UG/L	0	0%				0	18					47	U	49	U											48	U	48	U
Benzo(a)anthracene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Benzo(a)pyrene	UG/L	0	0%	GA	0		0	18					9	U	10	U											10	U	10	U
Benzo(b)fluoranthene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Benzo(ghi)perylene	UG/L	0.6	6%				1	18					0.6	J	10	U											10	U	10	U
Benzo(k)fluoranthene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Bis(2-Chloroethoxy)methane	UG/L	0	0%	GA	5	0	0	18					9	U	10	U											10	U	10	U
Bis(2-Chloroethyl)ether	UG/L	0	0%	GA	1	0	0	18					9	U	10	U											10	U	10	U
Bis(2-Chloroisopropyl)ether	UG/L	0	0%	GA	5	0	0	18					9	U	10	U											10	U	10	U
Bis(2-Ethylhexyl)phthalate	UG/L	11	6%	GA	5	1	1	18					9	U	10	U											10	U	10	U
Butylbenzylphthalate	UG/L	2	6%				1	18					9	U	10	U											10	U	10	U
Caprolactam	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Carbazole	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Chrysene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Dibenz(a,h)anthracene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Dibenzofuran	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Diethyl phthalate	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Dimethylphthalate	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Di-n-butylphthalate	UG/L	0	0%	GA	50	0	0	18					9	U	10	U											10	U	10	U
Di-n-octylphthalate	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Fluoranthene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Fluorene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	UJ
Hexachlorobenzene	UG/L	0	0%	GA	0.04	0	0	18					9	U	10	U											10	U	10	U
Hexachlorobutadiene	UG/L	0	0%	GA	0.5	0	0	18					9	U	10	U											10	U	10	U
Hexachlorocyclopentadiene	UG/L	0	0%	GA	5	0	0	18					42	U	44	U											43	U	43	U
Hexachloroethane	UG/L	0	0%	GA	5	0	0	18					9	U	10	U											10	U	10	U
Indeno(1,2,3-cd)pyrene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Isophorone	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Naphthalene	UG/L	2	6%				1	18					9	U	10	U											2	J	10	U
Nitrobenzene	UG/L	0	0%	GA	0.4	0	0	18					9	U	10	U											10	U	10	U
N-Nitroso-di-n-propylamine	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
N-Nitrosodiphenylamine	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Pentachlorophenol	UG/L	0	0%	GA	1	0	0	18					47	U	49	U											48	U	48	U
Phenanthrene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U
Phenol	UG/L	0	0%	GA	1	0	0	18					9	U	10	U											10	U	10	U
Pyrene	UG/L	0	0%				0	18					9	U	10	U											10	U	10	U

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Area	Loc ID	Matrix	Sample ID	Sample Depth Interval (FT)	Sample Date	QC Type	Study ID	Sample Round	Filtered	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
									SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25	SEAD-25
									MW25-3	MW25-3	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-8	MW25-9	MW25-9	MW25-9	MW25-9
									GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GROUNDWATER	GR
									25LM20115	25LM20121	25LM20003	25LM20012	25LM20037	25LM20047	25LM20059	25LM20092	25LM20103	25LM20004	25LM20013															
									8.8-8.8	5.23-5.23	0-0.1	0-0.1	0-0.1	0-0.1	0-0.1	4.41-4.41	4.25-4.25	0-0.1	0-0.1															
									3/18/2015	3/17/2016	1/31/2006	8/11/2006	3/4/2008	4/29/2009	1/13/2010	2/29/2012	5/7/2013	1/31/2006	8/9/2006															
									DU	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA															
									LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM	LTM															
									12	13	1	2	4	5	6	9	10	1	2															
									Total	Total	Total	Total	Total	Total	Total	Total	Total	Total	Total															
Inorganics																																		
Iron	UG/L	15,700	85%	GA	300	56	84	99	50	U	**320**		**329**	**J**	**667**		**349**		**620**		**408**		**411**	**J**	**4,200**		56.9	J	12	U
Sodium	UG/L	58,100	100%	GA	20,000	14	99	99	5,800		4,900		5,110		7,060	J	4,180		6,000		9,740		6,650		8,100		14,500		16,400	J
Wet Chemistry - MEE																																		
Chloride	MG/L	97.9	78%	GA	250	0	78	100	1.1		0.8		1.4		0.73	J	0.2	U	3.2		0.5	U	1.3	J	1	U	1.1		0.99	J
Ethane	UG/L	1.1	5%				5	104	0.55	U	0.55	U	2	U	2	U	1	U	1	U	0.16	U	0.58	U	0.81	U	2	U	2	U
Ethene	UG/L	4.6	5%				5	104	0.5	U	0.5	U	2	U	2	U	1	U	1	U	0.17	U	0.69	U	0.73	U	2	U	2	U
Methane	UG/L	170	57%				59	104	2		6.3		2	U	2	U	0.36	J	16		0.14	U	4.7	J	1.1	J	29		2	U
Nitrate	MG/L	6.4	68%	GA	10	0	45	66	0.69		0.028	J							0.05	U	0.05	UJ	0.017	J	0.041	J				
Nitrate Nitrogen	MG/L	1	45%				13	29					0.05	U	0.13		0.607	J									0.05	U	0.1	
Nitrate/Nitrite Nitrogen	MG/L	1	64%	GA	10	0	16	25									0.607				0.003	UJ								
Nitrite	MG/L	0.73	27%	GA	1	0	18	66	0.01	U	0.01	U							0.016		0.007	UJ	0.022	J	0.01	U				
Nitrite Nitrogen	MG/L	0.087	3%				1	29					0.05	U	0.05	U	0.01	UJ									0.05	U	0.05	U
Sulfate	MG/L	182	100%	GA	250	0	100	100	68		27		19.5		28.2		17.3		20.7		35.2	J	12	J	12		21.8		25.3	
Field Measurement - Hach Kit																																		
Conductivity	S/m	1.26	100%				90	90	0.686		0.689		0.494		0.72		0.427				0.342		0.462		0.506		0.535		0.718	
Conductivity (post)	S/m	0.844	100%				3	3																						
Conductivity (pre)	S/m	0.83	100%				3	3																						
Dissolved Oxygen	MG/L	12.6	100%				86	86	4.06		0.95		0.84		2.92		2.21				2.67		0.16		0.08		5.33		5.22	
Dissolved Oxygen (post)	MG/L	5.17	100%				3	3																						
Dissolved Oxygen (pre)	MG/L	5.36	100%				3	3																						
Nitrate Nitrogen	MG/L	0.5	100%				3	3																						
Nitrite Nitrogen	MG/L	0.5	100%				3	3																						
ORP	mV	259	100%				90	90	189		-15		-70		33.4		61				230		-133		-31		91		62.5	
ORP (post)	mV	197	100%				3	3																						
ORP (pre)	mV	193	100%				3	3																						
pH	Std units	8.37	100%				90	90	7.29		7.81		7.3		6.97		7.46				7.36		7.29		7.35		7.15		7.15	
pH (post)	Std units	7.38	100%				3	3																						
pH (pre)	Std units	7.38	100%				3	3																						
Sulfide	MG/L	1.04	89%				72	81	0		0.1		0.04		0.09		0.03		0.01		0.03		0.03		0.09		0.02		0.45	
Temperature	DEG C	26.55	100%				93	93	3.1		4.4		4.1		25.01		2.7				4.7		3.9		8.9		4.8		23.11	
Turbidity	NTU	195	100%				90	90	1.79		2.49		2.4		8.7		5.1				2.2		0.8		1.74		2.49		3.38	
Turbidity (post)	NTU	7.6	100%				2	2																						
Turbidity (pre)	NTU	5.7	100%				1	1																						

Notes:
1. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
2. Shading indicates concentration above cleanup goal.

U = compound was not detected
J = the reported value is an estimated concentration
UJ = the compound was not detected; the associated reporting limit is approximate
ND = Non-detect

SA = Sample
DU = Duplicate

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-25 MW25-9 GROUNDWATER 25LM20038 0-0.1 3/4/2008 SA LTM 4 Total Value	Qual	SEAD-25 MW25-9 GROUNDWATER 25LM20049 0-0.1 4/29/2009 SA LTM 5 Total Value	Qual	SEAD-25 MW25-9 GROUNDWATER 25LM20058 0-0.1 1/12/2010 SA LTM 6 Total Value	Qual	SEAD-25 MW25-9 GROUNDWATER 25LM20082 4.41-4.41 2/9/2011 SA LTM 8 Total Value	Qual	SEAD-25 MW25-9 GROUNDWATER 25LM20093 2.93-2.93 2/29/2012 SA LTM 9 Total Value	Qual	SEAD-25 MW25-9 GROUNDWATER 25LM20104 4.09-4.09 5/7/2013 SA LTM 10 Total Value	Qual	SEAD-25 MW25-9 GROUNDWATER 25LM20116 4.4-4.4 3/17/2015 SA LTM 12 Total Value	Qual	SEAD-25 MW25-9 GROUNDWATER 25LM20122 4.2-4.2 3/17/2016 SA LTM 13 Total Value	Qual
Volatile Organic Compounds																								
1,1,1-Trichloroethane	UG/L	0.62	2%	GA	5	0	2	107	1	U	1	U	0.32	U	0.2	UJ	0.2	U	0.5	UJ	0.37	U	0.37	U
1,1,2,2-Tetrachloroethane	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	0.09	U	0.38	UJ	0.38	U	0.18	U	0.62	U	0.62	U
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/L	0	0%	GA	5	0	0	102	1	U	1	U	0.4	U	0.31	U	0.31	UJ	0.5	U	0.36	U	0.36	U
1,1,2-Trichloroethane	UG/L	0	0%	GA	1	0	0	107	1	U	1	U	0.2	U	0.33	UJ	0.33	U	0.13	U	0.33	U	0.33	U
1,1-Dichloroethane	UG/L	3.5	9%	GA	5	0	10	107	1	U	1	U	0.14	U	0.21	UJ	0.21	U	0.25	U	0.38	U	0.38	U
1,1-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	0.37	U	0.35	UJ	0.35	U	0.11	U	0.36	U	0.36	U
1,2,4-Trichlorobenzene	UG/L	0	0%	GA	5	0	0	102	1	U	1	U	0.19	U	0.37	UJ	0.37	U	0.25	U	2.5	U	2.5	U
1,2,4-Trimethylbenzene	UG/L	0.45	7%	GA	5	0	2	30							0.19	UJ	0.19	U						
1,2-Dibromo-3-chloropropane	UG/L	0	0%	GA	0.04	0	0	102	2	U	2	U	0.43	U	0.5	U	0.5	U	0.44	U	1.1	U	1.1	U
1,2-Dibromoethane	UG/L	0	0%	GA	0.0006	0	0	107	1	U	1	U	0.18	U	0.22	UJ	0.22	U	0.25	U	0.44	U	0.44	U
1,2-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	1	U	0.4	U	0.15	UJ	0.15	U	0.21	U	0.37	U	0.37	U
1,2-Dichloroethane	UG/L	0.49	3%	GA	0.6	0	3	107	1	U	1	U	0.14	U	0.2	UJ	0.2	U	0.1	U	0.5	U	0.5	U
1,2-Dichloroethene (total)	UG/L	15	15%	GA	5	2	3	20							0.21	UJ	0.21	UJ						
1,2-Dichloropropane	UG/L	0	0%	GA	1	0	0	107	1	U	1	U	0.15	U	0.25	UJ	0.25	U	0.13	U	0.67	U	0.67	U
1,3,5-Trimethylbenzene	UG/L	0	0%	GA	5	0	0	30							0.2	UJ	0.2	U						
1,3-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	1	U	0.36	U	0.26	UJ	0.26	U	0.25	U	0.43	U	0.43	U
1,4-Dichlorobenzene	UG/L	0	0%	GA	3	0	0	102	1	U	1	U	0.34	U	0.24	UJ	0.24	U	0.28	U	0.46	U	0.46	U
Acetone	UG/L	11	7%				7	107	10	UJ	10	U	5	U	2.2	U	2.2	U	11	J	7	U	7	U
Benzene	UG/L	62	29%	GA	1	19	31	107	**2.3**		0.46	J	0.18	U	0.74	J	0.4	J	0.34	J	0.43	U	0.43	U
Bromodichloromethane	UG/L	0	0%	MCL	80	0	0	107	1	U	1	U	0.17	U	0.33	UJ	0.33	U	0.25	UJ	0.44	U	0.44	U
Bromoform	UG/L	0	0%	MCL	80	0	0	107	1	UJ	1	U	0.2	U	0.23	UJ	0.23	U	0.5	U	0.43	U	0.43	UJ
Carbon disulfide	UG/L	0.61	2%				2	107	1	U	1	U	0.36	U	0.25	U	0.25	U	0.6	U	1	U	1	U
Carbon tetrachloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	0.36	U	0.22	UJ	0.22	U	0.5	UJ	0.33	U	0.33	U
Chlorobenzene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	0.26	U	0.22	UJ	0.22	U	0.25	U	0.26	U	0.26	U
Chlorodibromomethane	UG/L	0	0%	MCL	80	0	0	107	1	U	1	U	0.11	U	0.3	UJ	0.3	U	0.1	U	0.32	U	0.32	U
Chloroethane	UG/L	0.67	2%	GA	5	0	2	107	2	U	1	U	0.21	U	0.55	U	0.55	U	2	U	2.5	U	2.5	U
Chloroform	UG/L	0.32	1%	GA	7	0	1	107	1	U	1	U	0.16	U	0.32	UJ	0.32	U	0.14	U	0.5	U	0.5	U
Cis-1,2-Dichloroethene	UG/L	19	14%	GA	5	4	15	107	1	U	1	U	0.14	U	0.21	UJ	0.21	U	0.15	U	0.41	U	0.41	U
Cis-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	1	U	1	U	0.14	U	0.19	UJ	0.19	UJ	0.11	U	0.4	U	0.4	U
Cyclohexane	UG/L	8.6	11%				11	102	1	U	1	U	0.14	U	0.31	UJ	0.31	U	0.25	U	0.39	U	0.39	U
Dichlorodifluoromethane	UG/L	0	0%	GA	5	0	0	82	1	UJ	1	U	0.18	U					0.25	U	0.6	UJ	0.6	U
Diisoproply Ether	UG/L	0	0%				0	20							0.21	U	0.21	U						
Ethyl benzene	UG/L	26	16%	GA	5	12	17	107	1	U	1	U	0.42	U	0.21	UJ	0.21	U	0.11	U	0.33	U	0.33	U
Isopropylbenzene	UG/L	2.6	9%	GA	5	0	10	107	1	U	1	U	0.34	U	0.23	UJ	0.23	U	0.1	U	0.35	U	0.35	U
Meta/Para Xylene	UG/L	19	11%	GA	5	4	7	61	0.43	J	2	U	0.81	U	0.59	UJ	0.59	U						
Methyl Acetate	UG/L	0	0%				0	102	10	U	2	U	0.48	U	0.53	U	0.53	U	0.19	U	1.8	U	1.8	U
Methyl bromide	UG/L	0	0%	GA	5	0	0	107	2	U	1	UJ	0.4	U	0.49	U	0.49	UJ	2	U	2.5	UJ	2.5	UJ
Methyl butyl ketone	UG/L	1.9	1%				1	107	5	U	5	U	0.4	U	1.7	U	1.7	U	1	U	2	U	2	U
Methyl chloride	UG/L	0	0%	GA	5	0	0	107	2	U	1	U	0.18	U	0.36	U	0.36	U	0.33	U	0.4	U	0.4	U
Methyl cyclohexane	UG/L	4.2	7%				7	102	1	U	1	U	0.16	U	0.3	U	0.3	U	0.1	U	0.43	U	0.43	U
Methyl ethyl ketone	UG/L	9	10%				11	107	5	U	5	U	1	U	1.3	U	1.3	U	1	U	3.4	U	3.4	U
Methyl isobutyl ketone	UG/L	0	0%				0	107	5	U	5	U	0.34	U	1.3	U	1.3	U	1	U	2.1	U	2.1	U
Methyl Tertbutyl Ether	UG/L	0	0%				0	107	1	U	1	U	0.13	U	0.36	U	0.36	U	0.2	U	0.3	U	0.3	U
Methylene chloride	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	0.13	U	1.1	U	1.1	U	1	U	2.5	U	2.5	U
Naphthalene	UG/L	0.23	4%				1	25							0.3	UJ	0.3	U						
n-Butylbenzene	UG/L	0	0%	GA	5	0	0	20							0.23	UJ	0.23	U						
Ortho Xylene	UG/L	6.4	8%	GA	5	3	5	61	1.5		1	U	0.4	U	0.25	UJ	0.25	U						
p-Isopropyltoluene	UG/L	0	0%	GA	5	0	0	10																
Propylbenzene	UG/L	0	0%	GA	5	0	0	10																
sec-Butylbenzene	UG/L	0	0%	GA	5	0	0	20							0.21	UJ	0.21	U						
Styrene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	0.36	U	0.23	UJ	0.23	U	0.11	U	0.27	U	0.27	U
tert-Butylbenzene	UG/L	0	0%	GA	5	0	0	20							0.31	UJ	0.31	U						
Tetrachloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	0.42	U	0.4	U	0.4	U	0.15	U	0.74	U	0.74	UJ
Toluene	UG/L	14	11%	GA	5	5	12	107	0.39	J	1	U	0.21	U	0.27	UJ	0.27	U	0.33	U	0.48	U	0.48	U
Total Xylenes	UG/L	62	11%	GA	5	3	7	66							0.25	UJ	0.25	U	0.2	U	0.23	U	0.23	U
Trans-1,2-Dichloroethene	UG/L	0	0%	GA	5	0	0	107	1	U	1	U	0.16	U	0.25	U	0.25	U	0.2	U	0.37	U	0.37	U
Trans-1,3-Dichloropropene	UG/L	0	0%	GA	0.4	0	0	107	1	U	1	U	0.17	U	0.2	UJ	0.2	UJ	0.21	UJ	0.42	U	0.42	U
Trichloroethene	UG/L	2	10%	GA	5	0	11	107	1	U	1	U	0.19	U	0.28	UJ	0.28	U	0.13	U	1.6		0.48	U
Trichlorofluoromethane	UG/L	0	0%	GA	5	0	0	102	1	U	1	U	0.16	U	0.24	U	0.24	U	0.25	U	0.42	U	0.42	U
Vinyl chloride	UG/L	2.6	6%	GA	2	2	6	107	1	U	1	U	0.22	U	0.25	U	0.25	U	0.18	U	0.5	U	0.5	U

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-9		MW25-9		MW25-9		MW25-9		MW25-9		MW25-9	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	
Sample ID									25LM20038		25LM20049		25LM20058		25LM20082		25LM20093		25LM20104		25LM20116		25LM20122	
Sample Depth Interval (FT)									0-0.1		0-0.1		0-0.1		4.41-4.41		2.93-2.93		4.09-4.09		4.4-4.4		4.2-4.2	
Sample Date									3/4/2008		4/29/2009		1/12/2010		2/9/2011		2/29/2012		5/7/2013		3/17/2015		3/17/2016	
QC Type									SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									4		5		6		8		9		10		12		13	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Semivolatile Organic Compounds																								
1,1'-Biphenyl	UG/L	0	0%	GA	5	0	0	18																
2,4,5-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18																
2,4,6-Trichlorophenol	UG/L	0	0%	GA	1	0	0	18																
2,4-Dichlorophenol	UG/L	0	0%	GA	5	0	0	18																
2,4-Dimethylphenol	UG/L	0	0%				0	18																
2,4-Dinitrophenol	UG/L	0	0%				0	18																
2,4-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18																
2,6-Dinitrotoluene	UG/L	0	0%	GA	5	0	0	18																
2-Chloronaphthalene	UG/L	0	0%				0	18																
2-Chlorophenol	UG/L	0	0%				0	18																
2-Methylnaphthalene	UG/L	0	0%				0	18																
2-Methylphenol	UG/L	0	0%				0	18																
2-Nitroaniline	UG/L	0	0%	GA	5	0	0	18																
2-Nitrophenol	UG/L	0	0%	GA	1	0	0	18																
3,3'-Dichlorobenzidine	UG/L	0	0%	GA	5	0	0	18																
3-Nitroaniline	UG/L	0	0%	GA	5	0	0	18																
4,6-Dinitro-2-methylphenol	UG/L	0	0%	GA	1	0	0	18																
4-Bromophenyl phenyl ether	UG/L	0	0%				0	18																
4-Chloro-3-methylphenol	UG/L	0	0%	GA	1	0	0	18																
4-Chloroaniline	UG/L	0	0%	GA	5	0	0	18																
4-Chlorophenyl phenyl ether	UG/L	0	0%				0	18																
4-Methylphenol	UG/L	0	0%				0	18																
4-Nitroaniline	UG/L	0	0%	GA	5	0	0	18																
4-Nitrophenol	UG/L	0	0%	GA	1	0	0	18																
Acenaphthene	UG/L	0.5	6%				1	18																
Acenaphthylene	UG/L	2	22%				4	18																
Acetophenone	UG/L	0	0%				0	18																
Anthracene	UG/L	1	6%				1	18																
Atrazine	UG/L	0	0%	GA	7.5	0	0	18																
Benzaldehyde	UG/L	0	0%				0	18																
Benzo(a)anthracene	UG/L	0	0%				0	18																
Benzo(a)pyrene	UG/L	0	0%	GA	0		0	18																
Benzo(b)fluoranthene	UG/L	0	0%				0	18																
Benzo(ghi)perylene	UG/L	0.6	6%				1	18																
Benzo(k)fluoranthene	UG/L	0	0%				0	18																
Bis(2-Chloroethoxy)methane	UG/L	0	0%	GA	5	0	0	18																
Bis(2-Chloroethyl)ether	UG/L	0	0%	GA	1	0	0	18																
Bis(2-Chloroisopropyl)ether	UG/L	0	0%	GA	5	0	0	18																
Bis(2-Ethylhexyl)phthalate	UG/L	11	6%	GA	5	1	1	18																
Butylbenzylphthalate	UG/L	2	6%				1	18																
Caprolactam	UG/L	0	0%				0	18																
Carbazole	UG/L	0	0%				0	18																
Chrysene	UG/L	0	0%				0	18																
Dibenz(a,h)anthracene	UG/L	0	0%				0	18																
Dibenzofuran	UG/L	0	0%				0	18																
Diethyl phthalate	UG/L	0	0%				0	18																
Dimethylphthalate	UG/L	0	0%				0	18																
Di-n-butylphthalate	UG/L	0	0%	GA	50	0	0	18																
Di-n-octylphthalate	UG/L	0	0%				0	18																
Fluoranthene	UG/L	0	0%				0	18																
Fluorene	UG/L	0	0%				0	18																
Hexachlorobenzene	UG/L	0	0%	GA	0.04	0	0	18																
Hexachlorobutadiene	UG/L	0	0%	GA	0.5	0	0	18																
Hexachlorocyclopentadiene	UG/L	0	0%	GA	5	0	0	18																
Hexachloroethane	UG/L	0	0%	GA	5	0	0	18																
Indeno(1,2,3-cd)pyrene	UG/L	0	0%				0	18																
Isophorone	UG/L	0	0%				0	18																
Naphthalene	UG/L	2	6%				1	18																
Nitrobenzene	UG/L	0	0%	GA	0.4	0	0	18																
N-Nitroso-di-n-propylamine	UG/L	0	0%				0	18																
N-Nitrosodiphenylamine	UG/L	0	0%				0	18																
Pentachlorophenol	UG/L	0	0%	GA	1	0	0	18																
Phenanthrene	UG/L	0	0%				0	18																
Phenol	UG/L	0	0%	GA	1	0	0	18																
Pyrene	UG/L	0	0%				0	18																

Appendix D
SEAD-25 Historic Groundwater Analytical Results
2016 Annual Long-Term Monitoring Report for SEAD-25
Seneca Army Depot Activity

Area									SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25		SEAD-25	
Loc ID									MW25-9		MW25-9		MW25-9		MW25-9		MW25-9		MW25-9	
Matrix									GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER		GROUNDWATER	
Sample ID									25LM20038		25LM20049		25LM20058		25LM20082		25LM20093		25LM20104		25LM20116		25LM20122	
Sample Depth Interval (FT)									0-0.1		0-0.1		0-0.1		4.41-4.41		2.93-2.93		4.09-4.09		4.4-4.4		4.2-4.2	
Sample Date									3/4/2008		4/29/2009		1/12/2010		2/9/2011		2/29/2012		5/7/2013		3/17/2015		3/17/2016	
QC Type									SA		SA		SA		SA		SA		SA		SA		SA	
Study ID									LTM		LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round									4		5		6		8		9		10		12		13	
Filtered									Total		Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Frequency of Detection	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																								
Iron	UG/L	15,700	85%	GA	300	56	84	99	100	U	**9,440**		**916**		**3,580**		**2,080**	**J**	**3,000**		92	J	20	J
Sodium	UG/L	58,100	100%	GA	20,000	14	99	99	8,380		**26,000**		16,500		**29,600**		**45,300**		**34,000**		14,000		15,000	
Wet Chemistry - MEE																								
Chloride	MG/L	97.9	78%	GA	250	0	78	100	0.2	U	2.7		0.5	U	1.6	J	0.55	J	1	U	2.3		0.44	J
Ethane	UG/L	1.1	5%				5	104	1	U	1	U	0.16	U	0.58	U	0.58	U	0.81	U	0.55	U	0.55	U
Ethene	UG/L	4.6	5%				5	104	1	U	1	U	0.17	U	0.69	U	0.69	U	0.73	U	0.5	U	0.5	U
Methane	UG/L	170	57%				59	104	2.4	J	3.5		0.14	U	5.4	J	4	J	0.45	U	0.8		0.69	
Nitrate	MG/L	6.4	68%	GA	10	0	45	66			0.05	U	0.05	UJ	0.0152	U	0.018	J	0.033	J	0.85		0.081	
Nitrate Nitrogen	MG/L	1	45%				13	29	0.05	UJ														
Nitrate/Nitrite Nitrogen	MG/L	1	64%	GA	10	0	16	25	0.05	U			0.003	UJ										
Nitrite	MG/L	0.73	27%	GA	1	0	18	66			0.01	U	0.007	UJ	0.00321	U	0.022	J	0.01	U	0.013	J	0.01	U
Nitrite Nitrogen	MG/L	0.087	3%				1	29	0.01	UJ														
Sulfate	MG/L	182	100%	GA	250	0	100	100	24.8		39.7		35.3	J	32	J	26	J	28		25		21	
Field Measurement - Hach Kit																								
Conductivity	S/m	1.26	100%				90	90	0.59				0.427				0.555		0.502		0.423		0.47	
Conductivity (post)	S/m	0.844	100%				3	3																
Conductivity (pre)	S/m	0.83	100%				3	3																
Dissolved Oxygen	MG/L	12.6	100%				86	86	2.02								1.77		0.16		10.97		0.83	
Dissolved Oxygen (post)	MG/L	5.17	100%				3	3																
Dissolved Oxygen (pre)	MG/L	5.36	100%				3	3																
Nitrate Nitrogen	MG/L	0.5	100%				3	3																
Nitrite Nitrogen	MG/L	0.5	100%				3	3																
ORP	mV	259	100%				90	90	99				-72				-129		-90		192		199	
ORP (post)	mV	197	100%				3	3																
ORP (pre)	mV	193	100%				3	3																
pH	Std units	8.37	100%				90	90	7.33				6.73				7.41		7.5		7.73		8.06	
pH (post)	Std units	7.38	100%				3	3																
pH (pre)	Std units	7.38	100%				3	3																
Sulfide	MG/L	1.04	89%				72	81	0.01	U	0.12		0.01						0.03		0.02		0.04	
Temperature	DEG C	26.55	100%				93	93	3.3				3.62				4.1		9.1		2.2		4.9	
Turbidity	NTU	195	100%				90	90	1.3				2.8				2.74		2.57		4.81		2.07	
Turbidity (post)	NTU	7.6	100%				2	2																
Turbidity (pre)	NTU	5.7	100%				1	1																

Notes:
1. Cleanup goal values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998).
2. Shading indicates concentration above cleanup goal.

U = compound was not detected
J = the reported value is an estimated concentration
UJ = the compound was not detected; the associated reporting limit is approximate
ND = Non-detect

SA = Sample
DU = Duplicate

APPENDIX E

LONG-TERM MONITORING EVENT 2016 DATA VALIDATION SHEETS

PROJECT NAME/NO. USACE - Seneca Army Depot SEAD-25 LTM Round 13
LAB: TestAmerica (TA)
SDG: 680-123117-1
FRACTION: TCL VOC (SW846 8260B)
MEDIA: GROUNDWATER
NUMBER OF SAMPLES: 9

CRITERIA	Did Analyses Meet all criteria as specified in the SOPS?	Region 2 Acceptable limits / criteria	Comments/Qualifying Actions	Qualifiers Added?
Data Completeness, Holding Times, Preservation, & Solids Percentage	Yes	Cooler temp < 10º. Samples holding time requirement < 14 days.	Coolers were received at 4.9-5.8°C on 3/17/16 and 3/18/16 by the laboratory. The samples were received in good condition based on the laboratory login report. The samples were analyzed within 14 days from sample collection.	No
System Monitoring Compounds	Yes	Recoveries within limits (70 - 130%) or laboratory established limits	All system monitoring compound recoveries were within the laboratory limits for all samples in this SDG.	No
Matrix Spike/Matrix Spike Duplicates and Laboratory Control Sample Recoveries	Yes	MS/MSD: 1 per 20 project samples. Recoveries within lab limits (or 70-130%). RPD < lab limit.	Sample 25LM20120 was designated for MS/MSD analyses. All MS/MSD precision and accuracy results were within criteria. All LCS/LCSD recoveries were within lab QC limits.	No
Blanks	Yes	Method blanks: 1 per 20 project samples. No TCL or TICs detected in MB, TB, or EB.	The laboratory method blank associated with the project samples did not contain target VOCs. The trip blanks 25LM00028 and 25LM00029 and the equipment blank 25LM00113 did not contain target VOCs.	No
GC/MS Instrument Performance Check	Yes	Performance check every 12 hours per instrument. Ion abundances normalized to m/z 95.	Checks were performed every 12 hours and the ion abundance was normalized to m/z 95.	No
TCL Analytes	Yes	RRT within 0.06 RRT units of standard RRT in CV.4. Relative intensities of characteristic ions within ± 30% of reference MS.	The major ions are present and the standard relative ion intensities generally agree within 20% for the primary quant ions for the compounds. All RTs within 0.06 RRT units of the standard RRT. No action was taken as the predominant ion intensities were generally consistent with the reference and calibration.	No
Tentatively Identified Compounds	N/A	No TCLs are listed as TIC. Ions in reference MS with relative intensity≥10% present in sample MS. TIC and "best match" standard relative ion intensities agree within ± 20%.	TICs were not reported for this SDG.	NA

PROJECT NAME/NO. USACE - Seneca Army Depot SEAD-25 LTM Round 13
LAB: TestAmerica (TA)
SDG: 680-123117-1
FRACTION: TCL VOC (SW846 8260B)
MEDIA: GROUNDWATER
NUMBER OF SAMPLES: 9

CRITERIA	Did Analyses Meet all criteria as specified in the SOPS?	Region 2 Acceptable limits / criteria	Comments/Qualifying Actions	Qualifiers Added?
Reported Quantitation Limits	Yes	Quantitation limits adjusted to reflect sample dilutions and moisture.	The lowest calibration standards were reported as reporting limits.	No
GC/MS Initial Calibration	Yes	%RSD ≤ 20%. Average RRFs > 0.050.	All initial calibrations associated with the project samples had %RSDs and mean RRFs within the criteria.	No
GC/MS Continuing Calibration	No	CCV performed for every 12 hours per instrument. %D ≤ 20%. RRFs ≥ 0.05.	The continuing calibration associated with samples collected on 3/16/16 had %D outlier for bromomethane (-23%D); and the continuing calibration associated with samples collected on 3/17/16 had %D outliers for bromomethane (-21.9%D), tetrachloroethene (20.8%D), and bromoform (28%D). Associated sample results were considered estimated with postive results qualified "J" and nondetected results qualified "UJ" for the affected samples.	Yes
Internal Standards	Yes	IS areas of samples & blank within (-50% to + 100%). RTs < 30 seconds.	Standard recovery area within the QC limits for all standards; and retention times were within 30 seconds of the standard for all samples that were used in this SDG.	No
Field Duplicate	Yes	All % RPD ≤ 50%?	Sample 25LM20120 was collected as the field duplicate sample of 25LM20119. All precision results were within criteria.	No

RT = Retention Time; %D = Percent Deviation; %RPD = Relative Percent Difference; %RSD = Percent Relative Standard Deviation; RRF = Relative Response Factor;
TCL = Target Compound List; TIC = Tentatively Identified Compound; CCV = Continuing Calibration Verification

PROJECT NAME/NO. USACE - Seneca Army Depot SEAD-25 LTM Round 13
SDG: 680-123117-1
LAB: TestAmerica (TA)
FRACTION: General Chemistry (sulfate and chloride - Method 300.0; nitrate and nitrite - Method 353.2)
MEDIA: Water

CRITERIA	Did Analyses Meet all criteria as specified in the SOPS?	If no, specify analysis IDs which do not meet criteria	Comments/Qualifying Actions	Qualifiers Added?
Data Completeness, Holding Times & Preservation	YES		Coolers were received at 4.9-5.8°C on 3/17/16 and 3/18/16 by the laboratory. The samples were received in good condition based on the laboratory login report.	NO
Calibration	YES		All instrument calibrations were within specified limits.	NO
Blanks	No		Initial calibration blanks and laboratory method blanks did not contain target anions. The continuing calibration blank associated with sample 25LM20120 contained nitrate at a concentration of 0.0245 J mg/L. Validation of this sample was not required. The equipment blank 25LM00113 did not contain target anions.	NO
Laboratory Control Sample	YES		LCS/LCSD recoveries met the specified criteria.	NO
Duplicates	YES		Sample 25LM20120 was collected as the field duplicate of 25LM20119. All precision results were less than 30%RPD.	NO
Spike Sample Analysis	YES		Sample 25LM20120 was designated for MS/MSD anlayses. All MS/MSD precision and accuracy results were within lab QC limits.	NO

PROJECT NAME/NO. USACE - Seneca Army Depot SEAD-25 LTM Round 13
SDG: 680-123117-1
FRACTION: Metals (iron and sodium)
LAB: Test America - Savannah
MEDIA: Groundwater

CRITERIA	Did Analyses Meet all criteria as specified in the SOPS?	If no, specify analysis IDs which do not meet criteria	Comments/Qualifying Actions	Qualifiers Added?
Data Completeness, Holding Times & Preservation	Yes		The cooler temperature was 4.9-5.8°C upon receipt by the laboratory. All samples were received in good condition based on the laboratory login report. Holding time met criteria.	No
Calibration	Yes		Calibrations available, taken every ten samples, and within recovery limits (90-110%) for metals. Initial calibration R2 >0.99.	No
Blanks (method blank, prep blank)	Yes		ICB, CCBs, and preparation blank did not contain iron and sodium. The rinsate blank 25LM00113 did not contain iron and sodium.	No
Interference Check Sample	Yes		Met requirements (80-120%) for iron and sodium.	No
CRQL Standard	Yes		CRQL Check Standards performed and within QC limit of 70-130%R.	No
Laboratory Control Sample	Yes		LCS results within limits (i.e., 80-120%) for iron and sodium.	No
Duplicates	Yes		Sample 25LM20120 was the field duplicate sample of 25LM20119. All precision results were less than 30%RPD.	No
Spike Sample Analysis	Yes		Sample 25LM20120 was designated for MS/MSD analyses. All MS/MSD precision and accuracy results were within criteria.	No
ICP Serial Dilution	YES		Sample 25LM20120 was designated for serial dilution analysis. All serial dilution results were less than 10%D.	No
Detection Limits	YES		IDLs available used as reporting limits. IDLs of iron and sodium are less than CRDLs.	No
ICP Linear Range	YES		All results within the ICP linear range.	No

PROJECT NAME/NO. USACE - Seneca Army Depot SEAD-25 LTM Round 13
LABORATORY: TestAmerica (TA)
SDG: 680-123117-1
MEDIA: Groundwater
FRACTION: Methane, Ethane, Ethene (USEPA approved SOP RSK-175)

CRITERIA	Did Analyses Meet all criteria as specified in the SOPS? Yes/No	Meet Criteria?	Comments	Qualifiers Added? Yes/No	Qualifying Actions
Data Package Completeness	All results forms and raw data, Cover Letter, and Case Narrative included? All samples in COC present? All notes in Case Narrative consistent with chemist's review of data package?	**Yes**		**No**	
Sample Conditions, Preservations, and Solids Percentage	Cooler temperature between 2ºC~6ºC? Record sample preservation and problems noted for sample conditions (e.g., bubbles?)	**Yes**	All samples received within one day of sample collection at 4.9-5.8ºC	**No**	
Holding Times	Samples met holding time requirement (non-preserved aqueous - 7 days; preserved aqueous - 14 days; non-aqueous - 14 days)	**Yes**		**No**	
Laboratory Control Sample (LCS)	LCS analyzed for every 20 project samples for corresponding matrix? LCS recoveries within laboratory limits (or 70~130% if not available)?	**Yes**		**No**	
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Was one MS/MD or one MS/MSD performed for every 20 project samples? Were recoveries within laboratory limits (or 70~130% if not available)?	**Yes**	Sample 25LM20120 was designated for MS/MSD analyses. All MS/MSD precision and accuracy results were within criteria.	**No**	

PROJECT NAME/NO. USACE - Seneca Army Depot SEAD-25 LTM Round 13
LABORATORY: TestAmerica (TA)
SDG: 680-123117-1
MEDIA: Groundwater
FRACTION: Methane, Ethane, Ethene (USEPA approved SOP RSK-175)

Blanks	1. Method blanks available for every 20 project samples? 2. Were trip blanks, rinsate blanks, and field blanks collected in accordance with QAPP (Table 16)? 3. No analytes should be detected in ICBs, CCBs, method blanks, trip blanks, or rinsate blanks. 4. Was chromatographic performance for laboratory blanks stable?	**No**	All laboratory blanks ND for MEE. The rinsate blank 25LM00113 contained methane at a concentration of 0.67 ug/L. Sample results were not affected and validation qualification was not required.	**No**	
Sample Result Verification	Were results verified with instrument raw data?	**Yes**		**No**	
Quantitation Limits	Were quantitation limits correctly calculated based on sample amount/volume and adjusted to reflect sample dilutions and, for soils, sample moisture?	**Yes**		**No**	
GC/MS Initial Calibration	1. ICVs analyzed at appropriate frequency with recoveries 90-110%R? 2. Curves linear for FID and TCD detectors?	**Yes**		**No**	
GC/MS Calibration Verification (CV)	1. Were CCV at the appropriate frequency with recoveries 90-110%R? 2. Were curves linear for the FID and TCD detectors?	**Yes**		**No**	
Field Duplicate	1. Was field duplicates collected for every 20 samples? 2. Were % RPDs ≤ 50% (soil) or 30% (aqueous) or difference ≤ 2RL (aqueous) or 4RL (soil) when one or both results<5RL?	**Yes**	Sample 25LM20120 was collected as the field duplicate sample of 25LM20119. All precision results were less than 30%RPD.	**No**	

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August 16, 2016

Ms. Amy Doss
U.S. Army Corps of Engineers
Engineering and Support Center, Huntsville
Attn: CEHNC-ED-CS-P
4820 University Square
Huntsville, AL 35816-1822

SUBJECT: Annual Report 2015 - Year 8 for the Abandoned Deactivation Furnace (SEAD-16) and the Active Deactivation Furnace (SEAD-17) Sites at Seneca Army Depot Activity in Romulus, NY; Contract W912DY-08-D-0003, Task Order 0015

Dear Ms. Doss:

Parsons Government Services, Inc. (Parsons) is pleased to submit the draft Annual Report 2015 - Year 8 for the Abandoned Deactivation Furnace (SEAD-16) and the Active Deactivation Furnace (SEAD-17) sites at the Seneca Army Depot Activity (SEDA) in Romulus, New York.

The recommendation enclosed in this Annual Report is to discontinue sampling, for the reasons stated. The Army concurs, but acknowledges that the requirement to continue sampling will continue until EPA and NYSDEC make the determination that it can be discontinued. The Army has contracted to continue the groundwater monitoring in the event that this requirement continues until the next Five Year Review (2021).

This work was performed in accordance with the Scope of Work (SOW) for Contract No. W912DY-08-D-0003, Task Order 0015.

Parsons appreciates the opportunity to provide you with the report for this work. Should you have any questions, please do not hesitate to call me at (617) 449-1405 to discuss them.

Sincerely,

Todd Heino, P.E., VP
Project Manager

Enclosures

cc: B. Frazier, USACE-Huntsville
K. Hoddinott, USACHPPM
R. Battaglia, USACE - NY District

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August 16, 2016

Mr. Julio Vazquez
USEPA Region II
Superfund Federal Facilities Section
290 Broadway, 18th Floor
New York, NY 10007-1866

Ms. Melissa Sweet
New York State Department of Environmental Conservation (NYSDEC)
Division of Environmental Remediation
625 Broadway, 12th Floor
Albany, NY 12233-7015

Mr. Mark Sergott
Bureau of Environmental Exposure Investigation
New York State Department of Health
Empire State Plaza Corning Tower, Room 1787
Albany, NY 12237

SUBJECT: **Annual Report 2015 - Year 8 for the Abandoned Deactivation Furnace (SEAD-16) and the Active Deactivation Furnace (SEAD-17) Sites at Seneca Army Depot Activity in Romulus, NY; EPA Site ID# NY0213820830 and NY Site ID# 8-50-006**

Dear Mr. Vazquez/Ms. Sweet/Mr. Sergott:

Parsons Government Services, Inc. (Parsons) is pleased to submit the draft Annual Report 2015 - Year 8 for the Abandoned Deactivation Furnace (SEAD-16) and the Active Deactivation Furnace (SEAD-17) sites at the Seneca Army Depot Activity (SEDA) in Romulus, New York (USEPA Site ID# NY0213820830 and NY Site ID# 8-50-006).

The recommendation enclosed in this Annual Report is to discontinue sampling, for the reasons stated. The Army concurs, but acknowledges that the requirement to continue sampling will continue until EPA and NYSDEC make the determination that it can be discontinued. The Army has contracted to continue the groundwater monitoring in the event that this requirement continues until the next Five Year Review (2021).

Parsons appreciates the opportunity to provide you with this report for this work. Should you have any questions, please do not hesitate to call me at (617) 449-1405 to discuss them.

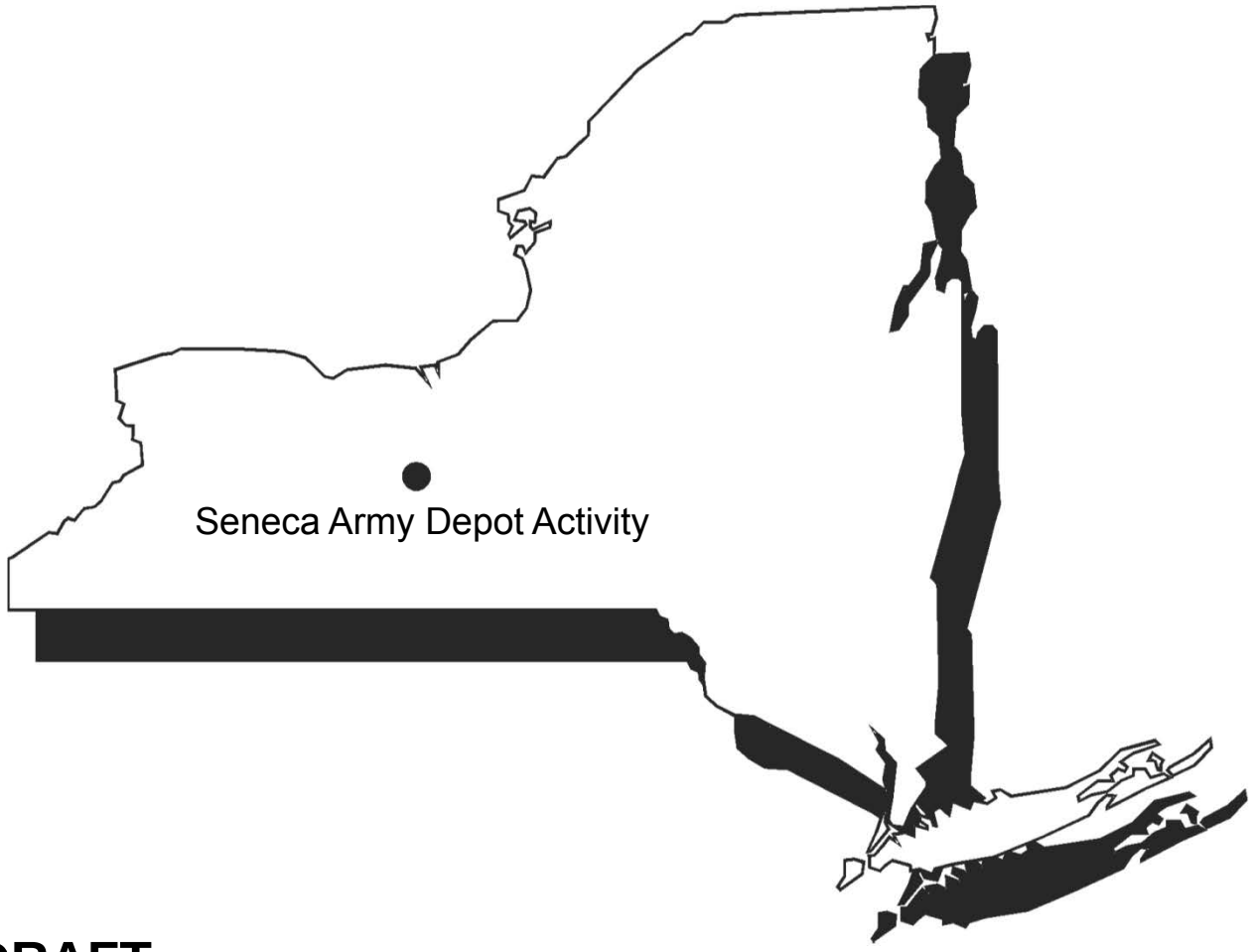
Sincerely,

Todd Heino, P.E., VP
Project Manager

Enclosures
cc: A. Doss, USACESCH; B. Frazier, USAESCH; K. Hoddinott, USACHPPM; R. Battaglia, USACE-NY

US Army, Engineering & Support Center
Huntsville, AL

Seneca Army Depot Activity
Romulus, NY



DRAFT
ANNUAL REPORT 2015 – YEAR 8
ABANDONED DEACTIVATION FURNACE (SEAD-16)
AND ACTIVE DEACTIVATION FURNACE (SEAD-17)
SENECA ARMY DEPOT ACTIVITY

Contract No. W912DY-08-D-0003
Task Order No. 0015
EPA Site ID# NY0213820830
NY Site ID# 8-50-006

PARSONS
August 2016

DRAFT

ANNUAL REPORT 2015 – YEAR 8

FOR THE ABANDONED DEACTIVATION FURNACE (SEAD-16)

AND THE ACTIVE DEACTIVATION FURNACE (SEAD-17)

SENECA ARMY DEPOT ACTIVITY, ROMULUS, NY

Prepared for:

U.S. ARMY CORPS OF ENGINEERS, ENGINEERING AND SUPPORT CENTER, HUNTSVILLE, ALABAMA

U.S. ARMY, CORPS OF ENGINEERS, NEW YORK DISTRICT
NEW YORK, NEW YORK

and

SENECA ARMY DEPOT ACTIVITY

ROMULUS, NY

Prepared by:

PARSONS
100 High Street
Boston, MA 02110

Contract Number W912DY-08-D-0003
Task Order 0015
EPA Site ID# NY0213820830
NY Site ID# 8-50-006

August 2016

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1.0 INTRODUCTION

This Draft Annual Report – Year 8 for the former Abandoned Deactivation Furnace (SEAD-16) and the former Active Deactivation Furnace (SEAD-17) sites at the Seneca Army Depot Activity (SEDA or the Depot) in Romulus, Seneca County, New York provides a review of annual groundwater monitoring data collected in December 2015, comparisons of the 2015 data to other pre- and post-remedial action (RA) groundwater sampling events, recommendations for future long-term monitoring (LTM) at SEAD-16 and SEAD-17, and the annual review of the effectiveness of the remedy implemented at the sites in 2007.

In accordance with the Record of Decision (ROD) for SEAD-16 and SEAD-17 (Parsons, 2006) and the *Remedial Design Work Plan and Design Report* (Parsons, 2007) (Final Work Plan), a RA was completed in August 2007 at SEAD-16 and SEAD-17 [the areas of concern (AOCs)]. The RA consisted of the excavation and disposal of soil, from both AOCs, which was contaminated with selected metals (antimony, arsenic, cadmium, copper, lead, mercury, thallium, and zinc) at levels above identified risk-based action levels. In addition, soil at SEAD-16 was also contaminated with polyaromatic hydrocarbons (PAHs) at concentrations in excess of risk-based action levels. The PAH impacted soil was excavated and was disposed of at a licensed landfill. The RA implemented at SEAD-16 and SEAD-17 is documented in the *Final Construction Completion Report for the Abandoned Deactivation Furnace (SEAD-16) and Active Deactivation Furnace (SEAD-17)* (Parsons, 2008). The RA at SEAD-16 involved the removal of approximately 1,862 cubic yards (cy) of soil which was impacted with metals and PAHs. The RA at SEAD-17 involved the removal of approximately 2,565 cy of metals-impacted soil.

The ROD for SEAD-16 and SEAD-17 also requires the implementation, maintenance, inspection, and periodic reporting of land use controls (LUCs) prohibiting use of the land at the AOCs for residential purposes and access to and use of groundwater until applicable cleanup standards are met. Applicable cleanup standards refer to the lowest enforceable standard associated with either the New York State Class GA (NYS Class GA) Ambient Water Quality Standards or United States Environmental Protection Agency (EPA) maximum contaminant levels (EPA MCLs). Once groundwater cleanup standards are achieved, the groundwater use restrictions may be eliminated upon approval of the EPA and the New York State Department of Environmental Conservation (NYSDEC). SEAD-16 and SEAD-17 are located within the Planned Industrial/Office Development and Warehousing (PID) area. The PID area has area-wide LUCs that prohibit the development and use of the property for residential housing, elementary and secondary schools, childcare facilities, and playgrounds; and, prohibits access to and use of groundwater until concentrations have been reduced to levels that allow for unlimited exposure and unrestricted use.

The Land Use Control Remedial Design (LUC RD) Addendum #4 identifies and implements the LUCs required by the SEAD-16 and SEAD-17 ROD at the identified AOCs, as well as other AOCs (SEADs 1, 2, 5, 59, 71, 121C, and 121I) in the PID area. The LUC objectives for SEAD-16 and SEAD-17 are to prevent access to or use of groundwater until New York State GA groundwater standards are achieved, and to prohibit residential housing, elementary and secondary schools, child care facilities and playground activities at the sites. Implementation of the LUCs at SEAD-16 and SEAD-17 may include lease restrictions, an environmental easement, deed restrictions, zoning, periodic certification, and a five-year review as is defined in the *Final Land Use Control Design for SEAD-27, 66, and 64A* (Army, 2006). The

LUC RD for SEAD-27, 66 and 64A is also known as the *LUC RD for the Planned Industrial/Office Development or Warehousing Area* that proposed the establishment of an area-wide set of land use restrictions for the PID/Warehouse Area to simplify institutional control implementation by having a single set of land use restrictions for the PID/Warehouse Area, which are consistent with its anticipated industrial land use. The periodic certification will be submitted to the NYSDEC and EPA to document that the LUCs at SEAD-16 and SEAD-17 are unchanged and that no activities have occurred that impair or violate the ability of the LUCs to protect public health and the environment.

Long-term groundwater monitoring is being performed at SEAD-16 and SEAD-17 as part of the post-closure monitoring and maintenance (PCMM) operations in accordance with the ROD and as outlined in the Final Work Plan (Parsons, 2007). LTM results are summarized in annual reports beginning in December 2007 (**Exhibit 1.1**). No LTM sampling event was conducted in 2011 due to budgetary constraints. This Year 8 report presents and discusses the results for the Year 8 LTM event which was conducted in December 2015.

Exhibit 1.1 – LTM and Inspection Summary

Round Number	Event	Date	Report Title
1	LTM	December 2007	Final Construction Completion Report for the Abandoned Deactivation Furnace (SEAD-16) and Active Deactivation Furnace (SEAD-17) (Parsons, 2008).
2	LTM	December 2008	Final Annual Report – Year 2 (Parsons, 2009)
3	LTM	November 2009	Final Annual Report – Year 3 (Parsons, 2010)
4	LTM	December 2010	Draft Final Annual Report – Year 4 (Parsons, 2013)
5	LTM	December 2012	Final Annual Report – Year 5 (Parsons, 2014a)
6	LTM	December 2013	Draft Annual Report – Year 6 (Parsons, 2014b)
7	LTM	December 2014	Draft Annual Report – Year 7 (Parsons, 2015)
8	LTM	December 2015	Draft Annual Report – Year 8

2.0 SITE BACKGROUND

2.1 Site Description

SEDA, a 10,587-acre former military facility located in Seneca County near Romulus, New York, is located between Seneca Lake and Cayuga Lake in Seneca County, and is bordered by New York State Highway 96 to the east, New York State Highway 96A to the west, and sparsely populated farmland to the north and south. The facility was wholly owned by the United States Government and was operated by the Department of the Army between 1941 and 2000; since 2000, portions of the Depot have been transferred to other parties for reuse. The primary mission of SEDA was the receipt, storage, maintenance, and supply of military items. A location map of SEDA is presented as **Figure 1**.

SEAD-16 and SEAD-17 are located in the east-central portion of the SEDA within the former ammunition storage area in an area where vehicular and pedestrian access is restricted. SEAD-16 and SEAD-17 are located in the portion of SEDA where land is presently designated for future PID uses. The locations of SEAD-16 and SEAD-17 are shown in **Figure 2**.

Both AOCs were historically used for the demilitarization of various small arms munitions. The munitions deactivation process involved heating the munitions in a rotating steel kiln. The heat would cause the munitions to detonate once the detonation temperature was reached. The byproducts produced during this detonation were then either swept out of the kiln through the stack or expelled from the kiln as bottom ash or debris.

SEAD-16, the former Abandoned Deactivation Furnace, was used from approximately 1945 until the mid 1960s when its use ceased and the site was vacated. The site consisted of 2.6 acres of fenced land with grasslands in the north, east, and west; a storage area for empty boxes and wooden debris located to the west; and an unpaved roadway in the south. Building S-311, which previously housed the deactivation furnace, was located at the approximate center of this area, and was demolished as part of the RA at SEAD-16. Documentation of demolition activities is presented in the *Building Cleaning and Building Demolition Completion Report* (Parsons, 2008). Building S-366, known as the Process Support Building, is located to the northeast of former Building S-311, and is currently unused and vacant. In addition to Building S-366, two sets of SEDA railroad tracks and utilities are presently on-site.

SEAD-17, the former Active Deactivation Furnace, was constructed to replace the Abandoned Deactivation Furnace at SEAD-16. However, SEAD-17 was inactive after 1989 as a result of Resource Conservation and Recovery Act (RCRA) permitting issues. SEAD-17 formerly consisted of the deactivation furnace, associated air pollution control equipment, and a support building (Building S-367), which were demolished or dismantled during the RA. Details and results of the demolition are documented in the *Building Cleaning and Building Demolition Completion Report* (Parsons, 2008). The former SEAD-17 deactivation furnace facility and support building were surrounded by a crushed shale road, beyond which lie grasslands. An unpaved gravel road to the north permits vehicular access to SEAD-17.

2.2 Site Hydrology

The hydrogeologic setting of SEAD-16 and SEAD-17 is described in detail in Sections 3.1.6 and 3.2.6, respectively, of the *Final Remedial Investigation (RI) Report at the Abandoned Deactivation Furnace (SEAD-16) and the Active Deactivation Furnace (SEAD-17)* (Parsons, 1999). A brief summary of the hydrogeologic conditions and chemical impacts, as described in the RI Report, is presented below.

2.2.1 SEAD-16

Three groundwater monitoring wells (MW16-1, MW16-2, and MW16-3) were installed as part of the Expanded Site Investigation (ESI) conducted at SEAD-16 in 1993/1994. Four additional groundwater monitoring wells (MW16-4, MW16-5, MW16-6, and MW16-7) were installed during the RI. As summarized in the *Final Construction Completion Report for the Abandoned Deactivation Furnace (SEAD-16) and Active Deactivation Furnace (SEAD-17)* (Parsons, 2008), MW16-3 was destroyed during the RA construction activities, and was not replaced as groundwater conditions on the southwest side of Building S-311 are adequately characterized by MW16-2 and MW16-5. The locations of the six existing groundwater monitoring wells and the former MW16-3 are shown on **Figure 3**.

Prior to the completion of the RA in August 2007, depth to groundwater was measured at SEAD-16 three times (April 1994, August 1996, and December 1996). Groundwater flow generally trends to the west based on previous subsurface investigations conducted at SEDA. Data from previous investigations suggest that a groundwater divide exists near, and approximately parallel to, Route 96 near Romulus, New York, indicating that the groundwater in the SEAD-16 area flows west. Based on available groundwater elevation data, it appears that there may be a regional groundwater high southwest of former Building S-311, which may contribute to local fluctuations in groundwater flow for the Site.

Horizontal hydraulic conductivities were determined for five wells screened in the till/weathered shale zone at SEAD-16. The saturated thickness in the till/weathered shale aquifer measured less than 2 feet when tested in September 1996. Hydraulic conductivity values for the shallow till/weathered shale aquifer ranged from 2.8×10^{-3} cm/sec to 2.5×10^{-2} cm/sec; the geometric mean was 7.3×10^{-3} cm/sec.

2.2.2 SEAD-17

Four groundwater monitoring wells (MW17-1, MW17-2, MW17-3, and MW17-4) were installed as part of the ESI conducted at SEAD-17. One additional groundwater monitoring well, MW17-5, was installed during the RI. The locations of the five groundwater monitoring wells installed at SEAD-17 are shown on **Figure 4**. Prior to the completion of the RA, depth to groundwater was measured at SEAD-17 in April 1994, August 1996, and December 1996 (the same time groundwater levels were measured at SEAD-16). Interpretation of groundwater elevation data indicates that groundwater flows to the southwest.

A horizontal hydraulic gradient of 0.01ft/ft was calculated between monitoring wells MW17-1 and MW17-3. Hydraulic conductivities were found to range from 2.9×10^{-3} cm/sec to 1.4×10^{-2} cm/sec.

2.3 Pre-Remedial Action Soil and Groundwater Conditions for SEAD-16

Pre-Remedial Action Soil Conditions

The primary historic constituents of concern (COCs) at SEAD-16 for soil included arsenic, copper, lead,

and zinc. The highest concentrations of soil contamination resulted from operations that were performed within and in close proximity to the former Abandoned Deactivation Furnace Building (S-311) and the Process Support Building (Bld. 366). Carcinogenic PAHs were detected in soils found at discrete locations within the AOC, with the highest concentrations detected in the surface soil samples collected adjacent to the northwestern corner of the former Abandoned Deactivation Furnace Building. Metals (antimony, copper, lead, mercury, and zinc) were found at concentrations greater than the site-specific cleanup goals in soil located in portions of the surrounding man-made drainage ditches.

Pre-Remedial Action Groundwater Conditions

Prior to completion of the RA, three rounds of low-flow groundwater sampling were conducted at SEAD-16, including one round in April 1994 as part of the ESI investigation activities, and two rounds in August and December 1996 as part of the RI activities. Compounds detected in the ESI and RI groundwater samples are presented in **Appendix A** (refer to the RI Report for complete groundwater analyses). Total metals were detected above either the applicable NYS Class GA standards or EPA MCLs. Concentrations exceeding applicable standards were less than or close to SEDA background concentrations, except for the exceedances of sodium. A summary of SEDA background groundwater data providing summary statistics (including maximum and average concentrations, the standard deviation for the collected data, and the frequency of detection) is provided in **Appendix B**. The Final Work Plan summarized that although metals were detected in the groundwater above their respective standards during previous sampling events, the groundwater was not impacted by site activities (Parsons, 2007). This conclusion was based on a comparison of results to the background groundwater data collected from unaffected parts of SEDA.

ESI and RI Data

Review of SEAD-16 data presented in the RI Report indicated that one or more concentrations measured for 14 metals (including arsenic, antimony, barium, beryllium, chromium, copper, iron, lead, manganese, mercury, nickel, selenium, sodium, and thallium) in 19 unfiltered groundwater samples collected during the ESI (performed in 1993/1994) and/or the RI (performed in 1999) exceeded NYS Class GA or EPA MCL standards in effect at the time of analysis. Of the 39 total instances where groundwater concentrations exceeded NYS Class GA or EPA MCL standards, 22 exceedances were associated with samples collected with peristaltic pumps (e.g., for the ESI sampling event) while the remaining 17 exceedances were found in samples collected using low-flow sampling with a bladder pump. Sample turbidities recorded during the RI sampling events were significantly lower than those recorded during the ESI sampling event, and thus are believed to be more representative of the water quality located at the site prior to the RA. Examination of the RI groundwater data shows that six metals were detected at concentrations in excess of NYS Class GA or EPA MCL standards in effect at the time of analysis:

- antimony (detected 2 times);
- iron (detected 5 times);
- lead (detected 1 time);

- manganese (detected 2 times);
- sodium (detected 3 times); and
- thallium (detected 4 times) EPA MCL.

Of these detections, antimony was detected at concentrations above the applicable NYS Class GA standard only in well MW16-3, with a maximum concentration of 12.3 µg/L. Iron was found at elevated concentrations in three wells: MW16-1 (at a maximum concentration of 2,400 J µg/L), MW16-2, and MW16-3. Lead was detected only in MW16-3 at a maximum concentration of 24.1 J µg/L; manganese was detected at elevated concentrations only in MW16-6 with a maximum level of 1,380 µg/L; sodium was detected in two wells (MW16-5 and MW16-6) with a maximum concentration of 409,000 µg/L detected at MW16-6; and thallium was detected in three wells including (MW16-2, MW16-5, and MW16-6), with a maximum concentration of 11 µg/L detected at MW16-6.

2.4 Pre-Remedial Action Soil and Groundwater Conditions for SEAD-17

Pre-Remedial Action Soil Conditions

The primary historic COCs in the soil at SEAD-17 were metals including antimony, arsenic, copper, lead, mercury, and zinc. The concentrations of metals were highest in samples collected closest to the location of the former Active Deactivation Furnace and its support building, particularly near the southwestern corner of the building.

Pre-Remedial Action Groundwater Conditions

Prior to the completion of the RA, three rounds of groundwater sampling were conducted at SEAD-17, concurrent with the sampling conducted at SEAD-16. Compounds detected in the groundwater samples collected during the low-flow sampling events in 1996 for SEAD-17 are presented in **Appendix A**. Total metals were detected at concentrations above the applicable NYS Class GA standards or EPA MCLs; however, except for sodium, these concentrations were lower than SEDA background metal concentrations (see SEDA background groundwater data summary in **Appendix B.**) The Final Work Plan summarized that, although metals had been detected in the groundwater above their respective standards during previous sampling events, the groundwater was not impacted by site activities. This conclusion is based on a comparison of results to groundwater data collected from non-impacted areas of SEDA.

ESI and RI Data

Review of SEAD-17 data presented in the RI Report indicated that one or more concentrations measured for four metals (i.e., iron, lead, sodium, and thallium) in 12 unfiltered groundwater samples exceeded NYS Class GA or EPA MCL standards in effect at the time of analysis. Of the 16 instances where groundwater concentrations exceeded the NYS Class GA or EPA MCL standards, 10 were associated with samples collected with a peristaltic pump (ESI sampling event) while the remaining six were found in samples collected using low-flow sampling with a bladder pump. As was indicated above for SEAD-16, sample turbidities recorded during the RI sampling events were lower than those recorded during the ESI sampling event, and thus the analytical results from the RI samples are believed to be more representative of the water quality present at SEAD-17. Examination of the RI groundwater data indicates

that only three metals (iron, sodium, and thallium) were detected at concentrations above NYS Class GA or EPA MCL standards in effect at the time of analysis. Of these detections, iron was detected at an elevated concentration in one well (MW17-1 at a concentration of 572 J µg/L); sodium was detected in two wells (MW17-3, at a maximum concentration of 30,100 µg/L, and at MW17-4); and thallium was detected in two wells (MW17-1 at a maximum concentration of 7.1 µg/L, and at MW17-5).

2.5 Remedial Action Summary

The selected remedy for SEAD-16 and SEAD-17 required the following:

- Excavation of soil impacted with metals and PAHs at concentrations greater than the site-specific cleanup standards;
- Stabilization of excavated soil exceeding the toxicity characteristic leaching procedure;
- Disposal of the material in an off-site landfill;
- Backfilling the excavated areas with clean backfill;
- Performing groundwater monitoring for select metals until groundwater concentrations do not exceed the applicable NYS Class GA or EPA MCL standards;
- Establishing and maintaining LUCs to prevent access to or use of groundwater and to prevent residential use of the land until cleanup standards are met; and
- Performing a review of the selected remedy every five years to evaluate if the remedy remains protective of the public health and the environment in accordance with Section 121(c) of the Comprehensive Environmental Remediation and Cleanup Liability Act (CERCLA).

The excavation of the impacted soil at SEAD-16 and SEAD-17 began on July 9, 2007 and was completed on August 2, 2007. Approximately 1,862 cy of impacted soil was removed from SEAD-16 and approximately 2,565 cy of impacted soil was removed from SEAD-17. The limit of the excavations performed at SEAD-16 is shown on **Figure 3** and for SEAD-17 on **Figure 4**.

Soil was excavated from both SEAD-16 and SEAD-17 until confirmatory soil samples collected from the sidewalls (when appropriate), the excavation floor, and the perimeter yielded analytical results below site-specific cleanup standards. The depth of excavation completed at SEAD-16 varied from approximately 1 to 3 feet below ground surface (bgs) and the excavation depth at SEAD-17 varied from approximately 1 to 2 feet bgs. The impacted soil from SEAD-16 and SEAD-17 was transported off-site and was disposed as non-hazardous material at the Ontario County Landfill in Flint, New York.

Deeper excavations at SEAD-16 and SEAD-17, including excavation areas surrounding the railroad tracks, were backfilled with clean bank-run gravel. SEAD-16 and SEAD-17 were graded to promote positive drainage. The areas at SEAD-17 that were vegetated prior to the RA were seeded to restore the vegetation. SEAD-16 was not seeded since it was not previously vegetated.

3.0 LONG TERM MONITORING RESULTS

3.1 Year 8 LTM Event

The Year 8 post-RA LTM event was conducted at SEAD-16 and SEAD-17 from December 19, 2015 through December 21, 2015. Groundwater samples were collected at SEAD-16 from six monitoring wells (MW16-1, MW16-2, MW16-4, MW16-5, MW16-6, and MW16-7) and from five monitoring wells (MW17-1, MW17-2, MW17-3, MW17-4, and MW17-5) located at SEAD-17. Field forms completed for the Year 8 sampling event are included in **Appendix C**. Groundwater data results for each LTM event are presented in **Appendix D,** and the laboratory analytical report for Year 8 is included as **Appendix E**. A discussion of data validation results is presented in **Appendix F**.

3.1.1 Year 8 Groundwater Elevations for SEAD-16 and SEAD-17

Prior to the collection of groundwater samples from each of the monitoring wells, groundwater elevation measurements were collected at each of the wells to be sampled. Groundwater elevation data for the Year 8 LTM event and historic data from past events are presented in **Table 1** and **Table 2** for SEAD-16 and SEAD-17, respectively. Groundwater elevations were measured on December 19, 2015 at SEAD-16 and SEAD-17.

Groundwater elevation data collected during previous investigations indicate that groundwater generally flows to the southwest at SEAD-16; however, historical groundwater elevation data also indicate that localized variation in groundwater flow direction may be due to higher groundwater elevations observed to the northeast and southwest of the former Building S-311. During the most recent (Year 8) LTM event, and similar with Years 4, 5, 6, and 7 LTM groundwater flow observations at SEAD-16, groundwater elevation data suggest that there is a groundwater low in the vicinity of the former Building S-311 location. The higher groundwater elevations to the northeast and southwest of the apparent groundwater low in the vicinity of Building S-311 result in two apparent local groundwater flow directions (to the southwest and northeast, respectively) (**Figure 5)**.

Based on the most recent elevation data (December 2015), groundwater at SEAD-17 appears to flow generally to the west-southwest, which is consistent with historical groundwater flow observations at SEAD-17 (**Figure 5)**.

3.1.2 Year 8 LTM Sample Collection

Samples for the Year 8 LTM event were collected using low-flow sampling techniques. A peristaltic pump was used in place of a bladder pump to collect the groundwater samples during this event due to winter weather conditions, including standing air temperatures below 32 degrees Fahrenheit (0 degrees Celsius). A peristaltic pump is recommended for freezing conditions since the bladder pump recharge cycle sequence allows water to freeze in the exposed portion of the sample tubing, which may inhibit sample collection efforts due to ice plugs forming in the tubing.

Sample collection, handling and custody, holding times, and field parameter collection procedures were conducted in accordance with the *Revised Final Sampling and Analysis Plan for Seneca Army Depot*

Activity (SAP) (Parsons, 2006c). Samples collected from the six SEAD-16 wells and the five SEAD-17 wells were submitted to TestAmerica (Savannah, GA) for the following analyses:

- Total Target Analyte List (TAL) metals, exclusive of mercury, by USEPA SW846 Method 6020; and
- Total mercury by USEPA SW846 7470A.

The TestAmerica Savannah, GA laboratory is certified by the Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) and the NELAC National Environmental Laboratory Accreditation Program (NELAP) for the above analyses/analytical methods for both potable and non-potable water.

Quality control (QC) samples, including one duplicate and one matrix spike/matrix spike duplicate (MS/MSD) pair, were collected at MW16-7. In the field, pH, oxidation-reduction potential (ORP), dissolved oxygen (DO), conductivity, temperature, and turbidity data were collected from each well during the purging cycle.

3.1.3 Year 8 LTM Sample Filtering

As documented in previous reports, there was the concern that elevated metal concentrations in SEAD-16 and SEAD-17 wells may be associated with higher groundwater turbidity values. With this in mind, both unfiltered and filtered samples were collected for the Year 3 through 7 LTM events: after the purging was complete, a sample was collected directly from the well as an unfiltered sample and then another sample was collected and filtered through a 0.45-micron membrane filter in the field and submitted as the filtered sample. Low turbidity values (< 5 Nephelometric Turbidity Units [NTU]) have been consistently observed in past rounds and during the Year 8 LTM event. As turbidity values were low (<5 NTU) during the Year 8 LTM event, filtered samples were determined to not be necessary (**Appendix C**).

3.1.4 Year 8 Groundwater Results for SEAD-16

A summary of metals detected in groundwater during the Year 8 LTM event for SEAD-16 is presented in **Table 3A**. Groundwater data results for each LTM event are presented in **Appendix D** and the laboratory analytical report for Year 8 is included as **Appendix E**. Data validation results are presented in **Appendix F**; sample 16LM20054 (MW16-7, parent sample) was found to be non-compliant for barium, calcium, potassium, magnesium, manganese, sodium, lead and antimony. When compared to the results from the duplicate sample taken at the same location, and to previous rounds, the concentrations are anomalously high. The concentrations of the parent sample were flagged as estimated during data validation and concentrations from the duplicate sample, instead of the parent sample, will be used in the analysis below. Data validation utilized the EPA Region 2 Standard Operating Procedures (SOPs) revised in March 2013.

Within SEAD-16, total concentrations of antimony, iron and sodium were detected above applicable NYS Class GA standards (**Table 3A**). Antimony (total) exceeded the NYS Class GA standard of 3 μg/L in one well (MW16-7). Antimony (total) was detected in four other wells (MW16-2, MW16-4, MW16-5, and MW16-6); however, the concentrations were estimated ("J" qualifier) and below the GA standard. Antimony was not detected in MW16-1.

Iron (total) exceeded the NYS Class GA standard (300 µg/L) in two wells. The highest concentration was detected in well MW16-6 (4,000 µg/L) and the other detection was in well MW16-5 (570 µg/L). The iron concentrations in the other four wells were below the GA standard.

The concentrations (710 and 4,120 µg/L) of the sum of iron and manganese (total) in wells MW16-5 and MW16-6 exceeded the combined NYS Class GA standard of 500 µg/L with the primary contributing metal being iron (total) (570 and 4,000 µg/L, respectively). Although manganese was detected in the groundwater samples collected from the SEAD-16 wells, it was not detected at concentrations above its NYS Class GA standard (300 µg/L) during the Year 8 LTM event.

Sodium (total) was detected at concentrations above the NYS Class GA standard (20,000 µg/L) in wells MW16-1, MW16-4, and MW16-7. The highest concentration was detected in well MW16-4 (250,000 µg/L). Sodium (total) exceedances were also found in both well MW16-1 (62,000 µg/L) and in well MW16-7 (23,000 J µg/L, respectively).

In summary, concentrations (total) of two select metals (antimony and sodium) continue to be detected in the groundwater at SEAD-16 at levels that exceed NYS Class GA standards. Iron (total) exceeded its NYS Class GA standard in two wells.

3.1.5 Year 8 Groundwater Results for SEAD-17

A summary of metals detected in the Year 8 groundwater samples event for SEAD-17 is presented in **Table 3B**. Groundwater analytical results for each LTM event are presented in **Appendix D** and the laboratory analytical report for Year 8 is included as **Appendix E**. A discussion of data validation results is presented in **Appendix F**; there were no non-compliance issues reported. Data validation utilized the EPA Region 2 SOPs revised in March 2013.

Antimony (total) did not exceed its NYS Class GA standard (3 µg/L) in any of the wells sampled at SEAD-17. Iron (total) was detected at a concentration (360 µg/L) above its NYS Class GA standard (300 µg/L) in one well (MW17-1). No other metals exceeded applicable groundwater standards in Year 8 at SEAD-17 (**Table 3B**).

3.1.6 LTM Groundwater Data Trends

An examination of the data trends from the Year 1 to 8 LTM events is provided for SEAD-16 and SEAD-17 in the following discussions. The LTM trends were examined to determine if the LTM results show: 1) an overall decreasing trend; 2) overall compliance with groundwater standards; and 3) their similarity to SEDA background values. Summaries of metal exceedances detected during the Year 8 groundwater monitoring event for SEAD-16 and SEAD-17 are provided in **Tables 3A** and **3B**, respectively. The data results for the Year 1 through Year 8 LTM events are included as **Appendix D**.

3.1.6.1 LTM Groundwater Trends for SEAD-16

During the eight years of LTM sampling at SEAD-16, five metals have exceeded NYS Class GA or EPA MCL standards: antimony, iron, lead, manganese, and sodium. The full LTM data set is provided in **Appendix D**.

Groundwater at three wells (MW16-2, MW16-4 and MW16-7) frequently had detections of antimony (total) above the NYS Class GA standard of 3 µg/L. In the most recent event, antimony only exceeded the NYS Class GA standard in one well (MW16-7). A plot of antimony concentration versus time illustrates that at MW16-7 antimony was detected above the standard in each event at concentrations ranging from 9.58 µg/L to 19 J µg/L (**Figure 6A**). The concentrations of antimony (total) detected at MW16-2 have fluctuated from just above the standard to a maximum concentration of 7.1 µg/L (Event 5); and, concentrations of antimony (total) at MW16-4 have varied from non-detect to a maximum of 6.3 µg/L (Event 3) (**Figure 6A**). The maximum concentrations at both MW16-2 and MW16-4 are below the SEDA average background concentration of 8.2 µg/L and in the past three rounds were below, or approximately equal to, the NYS Class GA standard. Examination of **Figure 6A** illustrates that the elevated concentrations of antimony above background and above the standard are isolated to MW16-7.

Lead (total) is not a persistent COC in any of the wells at SEAD-16 (**Figure 6B)**. Lead (total) has exceeded the EPA MCL twice during eight years of post-RA monitoring at MW16-7 during the first and second LTM sampling events. Since the last exceedance at MW16-7 in 2008, lead (total) concentrations have remained below the EPA MCL for the last six events. The plot in **Figure 6B** illustrates that with the exception of the noted spike of lead concentrations in events 1 and 2, the concentrations are below the standards, and lead is not a COC.

Exceedances of the NYS Class GA standard for iron (total) are predominantly in well MW16-5; however, all of the concentrations are below the SEDA background value (4,476 µg/L) (**Appendix B**). The highest concentrations of iron (total) detected in the groundwater at SEAD-16 are typically from well MW16-5. During Year 8, the iron (total) result at well MW16-6 was uncharacteristically higher than historical results from the wells at SEAD-16. This is interpreted as an anomaly in well MW16-5 for Year 8 and concentrations in this well are expected to return to historical averages. Iron concentrations over the course of LTM at SEAD-16 are presented on **Figure 6C**.

Manganese concentrations are historically below its NYS Class GA standard (300 µg/L). One exceedance (631 µg/L) of manganese was detected in well MW16-7 during Event 1.

Sodium is a persistent contaminant identified in SEAD-16 wells. It has been detected in every sample collected from the site. Sodium concentrations detected in the groundwater are currently higher than what was found prior to the RA. The concentrations are possibly affected by the known salt pile storage area that is operated by the Seneca County Highway Department (located approximately 1,000 feet upgradient to the east-northeast of SEAD-16). As identified on **Figure 5**, the groundwater east (upgradient) of SEAD-16 travels towards the southwest, from the salt pile storage area towards SEAD-16. In satellite photos of the area, the "Unnamed Dirt Road" that originates from the salt storage area and extends towards SEAD-16 appears to have a white coloration; the white coloration is likely due to salt residue from runoff emanating from the salt pile. Historically, the highest concentrations of sodium were found in well MW16-4; this well is the most directly in line with the suspected path of the salt. The location of the Seneca County Highway Department salt pile storage area is indicated on **Figure 5**. Sampling has not been conducted at the salt pile, or immediately downgradient of it, as it is not a CERCLA release; the Army does not plan on conducting any sampling in this location.

The trend over time in the LTM data shows that there is no evidence of an area-wide or expanding plume at SEAD-16. Antimony is a COC at one well, MW16-7; at all other wells, it is below the SEDA site-wide average background concentration, and fluctuating close to or below the NYS Class GA standard.

Lead is not considered a COC as all concentrations have been below the EPA MCL for the last six events. Iron is not considered a COC, as iron concentrations are common in the groundwater at Seneca, and the SEAD-16 iron groundwater concentrations are below SEDA site-wide background values. Sodium concentrations are not related to site activities and are likely a result of salt pile operations; sodium is not considered a COC.

3.1.6.2 LTM Groundwater Trends for SEAD-17

During the eight years of LTM sampling, five metals have exceeded NYS Class GA or EPA MCL standards including antimony (total), iron (total), lead (total), manganese (total), and sodium (total) (**Appendix D**). Historically, lead (total) and manganese (total) exceeded their applicable screening levels once and twice, respectively; sodium (total) exceeded its screening criterion in three wells. None of these three metals exceeded their respective criteria in Event 8. Lead (total), manganese (total), and sodium (total) are not persistent COCs at SEAD-17 and are therefore not discussed below.

Exceedances of the 3 µg/L NYS Class GA standard for antimony (total) are limited to well MW17-2, as illustrated in **Figure 6A**. The maximum concentration (4.4 J µg/L) reported for antimony (total) was detected in Year 5 from MW17-2. The concentrations of antimony (total) show a declining trend through time with detected concentrations from the last three monitoring events approximately equal to, or below, the NYS GA standard. All of the antimony concentrations detected during LTM have been below the SEDA background value for antimony (8.2 µg/L) (**Appendix D**).

Lead (total) is not a persistent COC in any of the wells at SEAD-17 (**Figure 6B)**. Lead (total) exceeded the EPA MCL once during eight years of post-RA monitoring at MW17-2 during the third LTM sampling event. Since the last exceedance, lead (total) concentrations have remained below the EPA MCL. The plot in **Figure 6B** illustrates that with the exception of the noted spike in concentration of lead in event 3, the concentrations are below the standards, and lead is not a COC.

Nine exceedances of the NYS Class GA standard for iron (total) were found in samples collected from four wells (MW17-1 with two exceedances, MW17-2 with two exceedances; MW17-3 with three exceedances; and MW17-4 with two exceedances) (**Appendix D**). The maximum concentration (25,500 J µg/L) of iron (total) was detected in well MW17-2 during the Year 3 LTM event. Except for the maximum detected concentration, all of the concentrations of iron have been below the SEDA background (4,476 µg/L). The concentrations of iron (total) during the course of the LTM is presented on **Figure 6D**.

Overall, post-RA LTM results indicate that groundwater quality at SEAD-17 is not impacted by historic operations conducted in this area. There are no trends associated with the elevated concentrations of sodium at SEAD-17 (Appendix D). These concentrations are estimated and, in general, return to the historical baseline condition at each well. Typically, sodium concentrations at SEAD-17 are below the Seneca background (**Appendix B**).

The SEAD-17 Year 8 data continues to support that the groundwater at SEAD-17 has not been impacted by metals released from the former Active Deactivation Furnace site. The most recent concentrations of antimony were below the NYS Class GA standard.

3.2 Routine Inspections of SEAD-16 and SEAD-17 Monitoring Wells

Observation of the wells at SEAD-16 and SEAD-17 during the Year 8 LTM event indicates that the wells located on the site are in acceptable condition. No obstructions were encountered in the wells at SEAD-16 and SEAD-17 during the Year 8 sampling event.

4.0 REMEDY EVALUATION

As discussed above in **Section 2.5**, approximately 4,427 cy of metal and PAH impacted soil were removed from SEAD-16 and SEAD-17 during the RA conducted in the summer of 2007. The impacted soil was removed to minimize or eliminate the migration of hazardous contaminants from soil to groundwater. Soil that exceeded the site-specific cleanup standards, as based on the confirmatory soil data, was removed from SEAD-16 and SEAD-17.

The long-term groundwater monitoring performed over eight years following the completion of the 2007 RA shows that the soil removal remedy has been effective in minimizing the migration of select metals from soil to groundwater. Pre-RA groundwater quality concerns associated with arsenic, barium, beryllium, chromium, copper, iron, lead, mercury, nickel and thallium have been eliminated, as each of these metals, with the exception of iron and lead, have not been detected in the groundwater at SEAD-16 in excess of the applicable NYS Class GA or EPA MCL standards since the RA was completed. Lead was found twice at levels in excess of the applicable EPA MCL, but these exceedances were confined to a single well (MW16-7) during the Year 1 and Year 2 post-RA LTM sampling events; lead exceedances in MW16-7 have not been detected during subsequent sampling events. While iron and manganese concentrations in excess of NYS Class GA groundwater quality standards are still present, these results appear to be partially affected by turbidity issues or are attributable to the regional groundwater quality, and are not attributable to site activities. Noted sodium exceedances found in the groundwater at SEAD-16 appear to originate from the salt storage area located upgradient of SEAD-16 which is operated by the Seneca County Highway Department and are not attributable to site activities. Antimony continues to be detected at concentrations above the applicable NYS Class GA standard, but these exceedances appear to be predominantly limited to two wells (MW16-2 and MW16-7) where concentrations have remained generally consistent since the RA was completed.

The groundwater quality at SEAD-17 has improved since the completion of the RA. The few noted groundwater quality exceedances for metals other than iron and manganese appear to be limited to the initial Year 1 or Year 2 post-RA sampling events or to a sample where a turbidity impact is suspected (e.g., the sample collected from MW17-2 during the Year 3 LTM event) and where groundwater quality has improved since the exceedances were reported. Although the concentrations of iron were identified at concentrations above the applicable NYS Class GA standards and the results are greater than what has been observed historically at the site, there is not sufficient trend information to indicate that there a significant change in groundwater conditions. Iron exceedances reported for SEAD-17 are isolated and are most likely attributable to regional groundwater quality and are not attributable to site activities. Historically (Events 1, 3, 5, and 7) within SEAD-17, antimony has exceeded the NYS Class GA standard in one well (MW17-2) in both unfiltered and filtered samples. All of the exceedances have been less than 1.5 µg/L over the NYS Class GA standard and the last two exceedances, in Events 5 and 7, the concentrations were estimated. Antimony was not detected over the NYS Class GA standard in the latest LTM event. Although antimony has limited exceedances over the NYS Class GA standard, there is no trend in these data or evidence to suggest that these concentrations are different than background (**Appendix B**).

The remedy for SEAD-16 and SEAD-17 includes the implementation and maintenance of LUCs consisting of:

- Prevention of residential housing, elementary and secondary schools, childcare facilities and playground activities; and
- Prevention of access to or uses of the groundwater until concentrations are below the NYS Class GA Groundwater or EPA MCL standards.

As part of the LTM program, SEAD-16 and SEAD-17 were inspected to determine if the LUCs are being maintained. During the Year 8 event, it was confirmed that no residential housing, elementary and/or secondary schools, childcare facilities, or playgrounds have been constructed or established in these AOCs, and no access to or use of groundwater, beyond that which is gained by the existing monitoring well network, was evident at either SEAD-16 or SEAD-17. Access to and use of the groundwater is restricted at the AOC under the terms of the ROD and is not being used as a potable water source. A non-groundwater sourced municipal water supply is available for the Depot and includes the PID area. The groundwater access/use restriction will remain in effect at the PID and SEAD-16/17 until select metal concentrations in groundwater have been reduced to levels below applicable NYS Class GA and EPA MCL standards and until data demonstrating acceptable groundwater quality in the AOC is provided to and approved by the applicable regulatory agencies.

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

- The soil excavation remedy at SEAD-16 and SEAD-17 was an effective method for controlling, and in some cases eliminating, the migration of select metals from soil to groundwater based on the evaluation of the results of the eight post-RA LTM sampling events. Trends demonstrate that the remedial action performed did not adversely impact groundwater.
- There is no ongoing treatment process at either site to continue monitoring for concentration reductions.
- Post-remediation groundwater monitoring results indicate that there was a limited impact on the groundwater at SEAD-16/17. Iron, lead, and sodium were detected above groundwater standards in a limited number of wells; however, they currently are not considered COCs as they are below SEDA background levels and/or have not been detected above guidance values in the past several events.
- Antimony is a COC in one well, MW16-7; the concentrations at this well are stable.
- Antimony is not migrating, as evidenced by absence of increasing antimony concentrations in other wells.
- Groundwater use is prohibited by the area-wide LUC and an alternate potable water source is available. The land use and groundwater use restrictions imposed at SEAD-16 and SEAD-17 are maintained as part of both the approved RODs for SEAD 16/17 and the larger Planned Industrial/Office or Warehousing Area ("PID Area") (Parsons, 2004; 2006). There are no signs of unauthorized use or access to the AOCs.

5.2 Recommendations

Based on the current area-wide LUC prohibiting the use of groundwater within the PID Area (includes SEADs 16/17), the Army recommends concluding LTM at these sites because there is no planned future use of the groundwater. The wells will not be decommissioned at this time and sampling at these sites may take place in the future if the need arises (e.g., emerging contaminants, decisions during the 2021 5 Year Review). Annual LUC inspections will continue to insure that the groundwater is not accessed.

6.0 REFERENCES

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TABLES

Table 1
SEAD-16 - Groundwater Table Elevations Summary
Draft Annual Report - SEAD-16 and SEAD-17
Seneca Army Depot Activity

Pre-Remedial Action Groundwater Elevation Data

Monitoring Well	Top of PVC Elevation [1] (feet)	April 4, 1994		August 27, 1996		December 6, 1996	
		Depth to Water (feet)	Water Table Elevation (feet)	Depth to Water (feet)	Water Table Elevation (feet)	Depth to Water (feet)	Water Table Elevation (feet)
MW 16-1	735.54	3.52	732.02	6.45	729.09	3.25	732.29
MW 16-2	734.56	3.65	730.91	4.50	730.06	3.71	730.85
MW 16-3	735.48	4.60	730.88	5.43	730.05	4.64	730.84
MW 16-4	733.93	NA	NA	4.83	729.10	2.93	731.00
MW 16-5	733.40	NA	NA	4.76	728.64	2.20	731.20
MW 16-6	733.56	NA	NA	4.54	729.02	2.90	730.66
MW 16-7	734.42	NA	NA	5.06	729.36	4.23	730.19

Post-Remedial Action Groundwater Elevation Data

Monitoring Well	Top of PVC Elevation [1] (feet)	December 20, 2007		2008 Top of PVC Elevation [4, 5] (feet)	December 9, 2008		November 13, 2009		December 13, 2010	
		Depth to Water (feet)	Water Table Elevation (feet)		Depth to Water (feet)	Water Table Elevation [4,5] (feet)	Depth to Water (feet)	Water Table Elevation [5] (feet)	Depth to Water (feet)	Water Table Elevation [5] (feet)
MW 16-1	735.54	4.25	731.29	735.54	4.28	731.26	5.76	729.78	3.16	732.38
MW 16-2	734.56	4.20	730.36	733.48	4.20	729.28	4.35	729.13	4.08	729.40
MW 16-3	735.48	NA	NA	735.48	NA	NA	NA	NA	NA	NA
MW 16-4	733.93	3.00	730.93	733.93	3.42	730.51	3.91	730.02	2.78	731.15
MW 16-5	733.40	1.90	731.50	735.82	3.32	732.50	3.10	732.72	1.68	734.14
MW 16-6	733.56	2.66	730.90	733.56	3.47	730.09	3.68	729.88	2.53	731.03
MW 16-7	734.42	4.45	729.97	734.42	4.63	729.79	4.75	729.67	4.41	730.01

Monitoring Well	2012 Top of PVC Elevation [6] (feet)	December 10, 2012		December 9, 2013		December 15, 2014		December 19, 2015	
		Depth to Water (feet)	Water Table Elevation [6] (feet)	Depth to Water (feet)	Water Table Elevation [6] (feet)	Depth to Water (feet)	Water Table Elevation [6] (feet)	Depth to Water (feet)	Water Table Elevation [6] (feet)
MW 16-1	735.53	3.15	732.38	2.94	732.59	2.96	732.57	3.69	731.84
MW 16-2	734.86	4.08	730.78	4.18	730.68	3.8	731.06	3.33	731.53
MW 16-3	NA	NA	NA	NA	NA	NA	NA	NA	NA
MW 16-4	734.51	2.71	731.80	2.64	731.87	2.66	731.85	3.03	731.48
MW 16-5	735.36	1.63	733.73	2.26	733.10	1.64	733.72	2.2	733.16
MW 16-6	734.25	2.37	731.88	2.65	731.60	2.33	731.92	2.68	731.57
MW 16-7	734.96	4.28	730.68	4.38	730.58	4.08	730.88	3.52	731.44

(1) Elevations are relative to the North American Vertical Datum (NAVD) 1988.
(2) April 4, 1994 data were collected as a part of the ESI and August 1996 and December 1996 were collected during the Remedial Investigation phase.
(3) Monitoring well MW16-3 was destroyed during the remedial action conducted at SEAD-16.
(4) PVC riser pipe for wells MW16-2 and MW16-5 was necessary to be cut during December 2008 sampling event due to the PVC preventing the metal casing lid from opening.
(5) MW16-2 and MW16-5 were re-surveyed in Dec 2008 and this data was used for water table elevation calculations for December 9, 2008 through December 13, 2010. MW16-2 Top of PVC elevation is 733.48 ft, and MW16-5 Top of PVC elevation is 735.82 ft.
(6) Wells were re-surveyed with GPS RTK equipment in November 2012. New ground surface and top of the PVC elevations were used for the December 2012 water table elevation calculation.
NA = Not Available.

Table 2
SEAD-17 - Groundwater Table Elevations Summary
Draft Annual Report - SEAD-16 and SEAD-17
Seneca Army Depot Activity

Pre-Remedial Action Groundwater Elevation Data

Monitoring Well	2008 Top of PVC Elevation	Top of PVC Elevation [1] (feet)	April 4, 1994		August 29, 1996		December 6, 1996	
			Depth to Water (feet)	Water Table Elevation (feet)	Depth to Water (feet)	Water Table Elevation (feet)	Depth to Water (feet)	Water Table Elevation (feet)
MW 17-1	732.625	736.30	2.80	733.50	7.64	728.66	3.01	733.29
MW 17-2		733.75	3.19	730.56	7.24	726.51	3.45	730.30
MW 17-3		732.15	2.38	729.77	7.14	725.01	2.47	729.68
MW 17-4		734.59	3.00	731.59	7.23	727.36	3.13	731.46
MW 17-5		733.58	NA	NA	6.92	726.66	2.65	730.93

Post Remedial Action Groundwater Elevation Data

Monitoring Well	Top of PVC Elevation [1] (feet)	December 19, 2007		2008 Top of PVC Elevation	December 9, 2008		November 11, 2009		December 13, 2010	
		Depth to Water (feet)	Water Table Elevation (feet)		Depth to Water (feet)	Water Table Elevation [3,4] (feet)	Depth to Water (feet)	Water Table Elevation [4] (feet)	Depth to Water (feet)	Water Table Elevation [4] (feet)
MW 17-1	736.30	3.33	732.97	736.30	4.25	732.05	5.60	730.70	3.32	732.98
MW 17-2	733.75	3.31	730.44	733.75	4.07	729.68	5.27	728.48	2.2	731.55
MW 17-3	732.15	2.67	729.48	732.625	3.96	728.67	6.15	726.48	2.51	730.12
MW 17-4	734.59	3.40	731.19	734.59	4.05	730.54	5.75	728.84	3.4	731.19
MW 17-5	733.58	2.90	730.68	733.58	3.46	730.12	4.65	728.93	2.79	730.79

Monitoring Well	2012 Top of PVC Elevation [5] (feet)	December 10, 2012		December 9, 2013		December 15, 2014		December 19, 2015	
		Depth to Water (feet)	Water Table Elevation [5] (feet)	Depth to Water (feet)	Water Table Elevation [5] (feet)	Depth to Water (feet)	Water Table Elevation [5] (feet)	Depth to Water (feet)	Water Table Elevation [5] (feet)
MW 17-1	736.39	3.19	733.20	3.52	732.87	3.26	733.13	3.55	732.84
MW 17-2	733.65	2.79	730.86	3.15	730.50	2.77	730.88	3.5	730.15
MW 17-3	732.05	2.4	729.65	2.73	729.32	2.38	729.67	3.73	728.32
MW 17-4	734.62	3.18	731.44	3.2	731.42	3.22	731.40	3.28	731.34
MW 17-5	734.12	2.64	731.48	2.79	731.33	2.64	731.48	2.96	731.16

Notes:
(1) Elevations are relative to the North American Vertical Datum (NAVD) 1988.
(2) April 4, 1994 data were collected as a part of the ESI and August 1996 and December 1996 were collected during the Remedial Investigation Phase.
(3) PVC riser pipe for MW17-3 was necessary to be cut during December 2008 sampling event due to the PVC preventing the metal casing lid from opening.
(4) MW17-3 was re-surveyed in December 2008 and this data was used for water table elevation calculations for December 9, 2008 through December 13, 2010. MW17-3 Top of PVC elevation is 732.63 ft.
(5) Wells were re-surveyed with GPS RTK equipment in November 2012. New ground surface and top of the PVC elevations were used for December 2012 water table elevation calculation.
NA = Not Available.

Table 3A
SEAD-16 Detected Groundwater Compounds
Draft Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area				SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16	
Loc ID				MW16-1		MW16-2		MW16-4		MW16-5		MW16-6		MW16-7		MW16-7	
Matrix				GW		GW		GW		GW		GW		GW		GW	
Sample ID				16LM20049		16LM20050		16LM20051		16LM20052		16LM20053		16LM20054		16LM20055	
Sample Date				12/20/2015		12/19/2015		12/20/2015		12/19/2015		12/19/2015		12/19/2015		12/19/2015	
QC Type				SA		SA		SA		SA		SA		SA		DU	
Study ID				LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round				8		8		8		8		8		8		8	
Filtered				Total		Total		Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Criteria Level	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Aluminum	UG/L	2,400		44	J	58	J	18	U	31	J	2,400		140	J	36	J
Antimony	UG/L	120	3	0.5	U	2.1	J	2	J	0.75	J	1	J	**120**	**J**	**19**	**J**
Arsenic	UG/L	1.9	10	1.5	U	1.5	U	1.5	U	1.5	U	1.9	J	7.5	U	1.5	U
Barium	UG/L	600	1,000	81		94		140		41		73		600	J	130	J
Cadmium	UG/L	0.34	5	0.15	U	0.15	U	0.34	J	0.15	U	0.33	J	0.15	U	0.15	U
Calcium	UG/L	510,000		120,000		130,000		160,000		110,000		80,000		510,000	J	110,000	J
Chromium	UG/L	4.6	50	3	J	1.6	U	1.6	U	1.6	U	4.6	J	8	U	1.6	U
Cobalt	UG/L	1.6		0.12	J	0.68		0.28	J	0.12	U	1.6		0.6	U	0.12	J
Copper	UG/L	21	200	1.7	U	3	J	6.8		1.7	U	6.3		21	J	4.2	J
Iron	UG/L	4,000	300	68	J	130		33	J	**570**		**4,000**		**370**	**J**	62	J
Iron+Manganese	UG/L	4,120	500	76.7	J	193		85	J	**710**		**4120**		396	J	69.4	J
Lead	UG/L	48	15	0.98	U	2.9		1.1	J	0.98	U	2.2	J	**48**	**J**	10	J
Magnesium	UG/L	98,000		19,000		13,000		25,000		10,000		8,300		98,000	J	20,000	J
Manganese	UG/L	140	300	8.7		63		52		140		120		26	J	7.4	J
Nickel	UG/L	5.1	100	3.8	J	2.6	J	3.7	J	2.3	J	5.1		9.5	U	1.9	U
Potassium	UG/L	15,000		1,000		1,900		1,900		2,500		2,600		15,000	J	3,600	J
Selenium	UG/L	1.1	10	1	U	1	U	1	U	1	U	1.1	J	5	U	1	U
Sodium	UG/L	250,000	20,000	**62,000**		11,000		**250,000**		1,800		10,000		**89,000**	**J**	**23,000**	**J**
Zinc	UG/L	18		9.6	U	17	J	16	J	9.6	U	18	J	48	U	9.6	U

Notes:

1. The criteria values (where available) are NYS Class GA Groundwater Standards (TOGS 1.1.1, June 1998) and EPA Maximum Contamination Limit (MCL), Source http://www.epa.gov/safewater/mcl.html#inorganic.html
2. Shading indicates a concentration above the GA or MCL groundwater standard.
3. A blank in the Criteria Level column indicates no standard established for that compound.

U = compound was not detected
J = the reported value is an estimated cocentration
SA = Sample
DU = Duplicate Sample

Table 3B
SEAD 17 Detected Groundwater Compounds
Draft Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

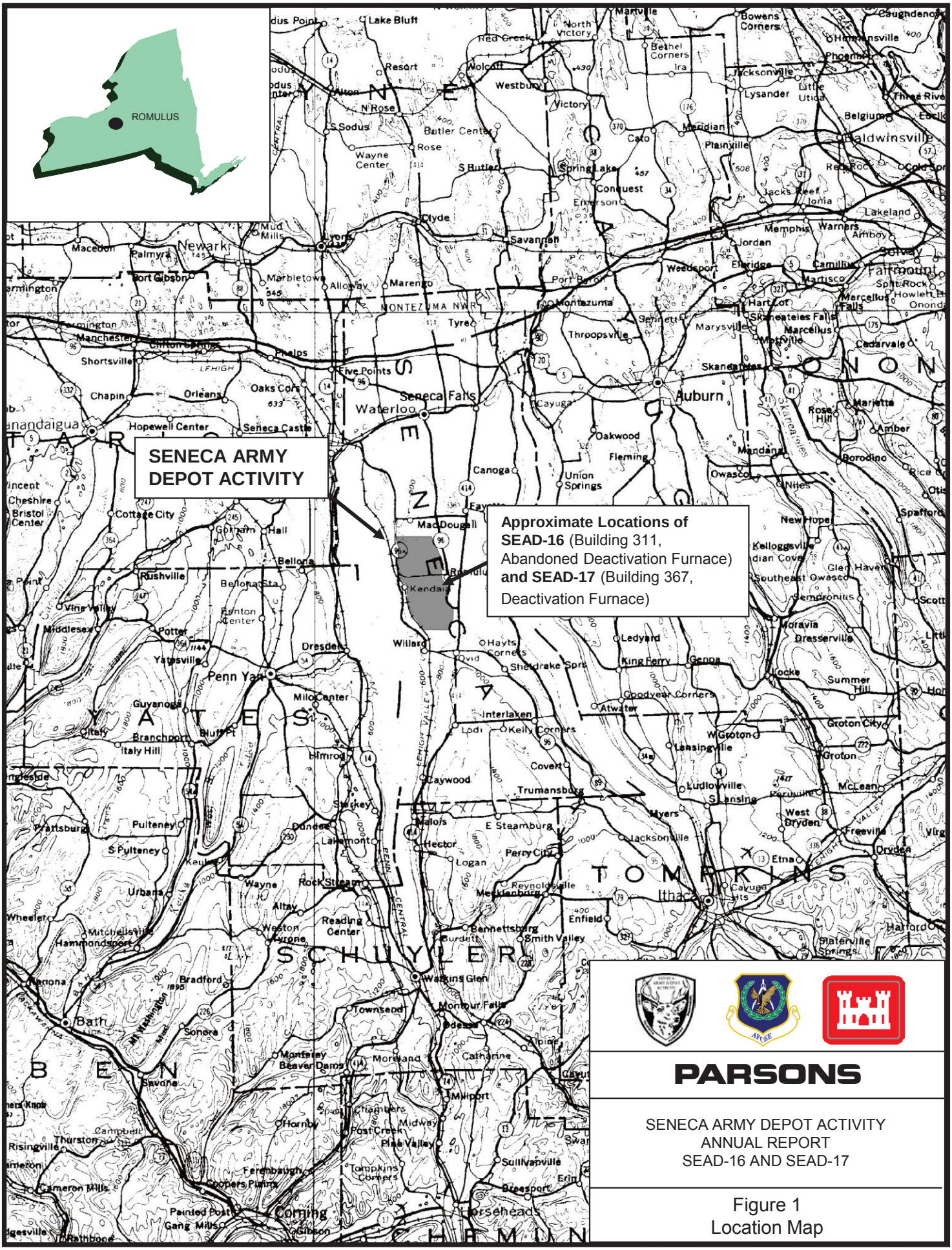
Area				SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17	
Loc ID				MW17-1		MW17-2		MW17-3		MW17-4		MW17-5	
Matrix				GW		GW		GW		GW		GW	
Sample ID				17LM20035		17LM20036		17LM20037		17LM20038		17LM20039	
Sample Date				12/21/2015		12/20/2015		12/20/2015		12/21/2015		12/20/2015	
QC Type				SA		SA		SA		SA		SA	
Study ID				LTM		LTM		LTM		LTM		LTM	
Sample Round				8		8		8		8		8	
Filtered				Total		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Criteria Level	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Aluminum	UG/L	31		18	J	19	J	31	J	18	U	18	U
Antimony	UG/L	0.63	3	0.5	U	0.63	J	0.5	U	0.56	J	0.5	U
Barium	UG/L	86	1,000	70		66		51		29		86	
Calcium	UG/L	160,000		98,000		160,000		100,000		80,000		100,000	
Cobalt	UG/L	1.1		0.3	J	0.42	J	0.12	U	1.1		0.14	J
Copper	UG/L	2.4	200	1.7	U	2.4	J	1.7	U	1.7	U	1.7	U
Iron	UG/L	360	300	**360**		140		43	J	59	J	43	J
Iron+Manganese	UG/L	449	500	449		175		44.8	J	158	J	48.8	J
Lead	UG/L	1.5	15	0.98	U	0.98	U	0.98	U	1.5	J	0.98	U
Magnesium	UG/L	19,000		19,000		16,000		11,000		11,000		17,000	
Manganese	UG/L	99	300	89		35		1.8	U	99		5.8	
Nickel	UG/L	2.1	100	1.9	U	1.9	U	1.9	U	2.1	J	1.9	U
Potassium	UG/L	1,600		520	J	1,600		810	J	500	J	1,300	
Sodium	UG/L	12,000	20,000	6,400		12,000		8,400		6,000		5,800	
Zinc	UG/L	27		9.6	U	26		27		9.6	U	9.6	U

Notes:

1. The criteria values (where available) are NYS Class GA Groundwater Standards (TOGS 1.1.1, June 1998)
and EPA Maximum Contamination Limit (MCL), Source http://www.epa.gov/safewater/mcl.html#inorganic.html
2. Shading indicates a concentration above the GA or MCL groundwater standard.
3. A blank in the Criteria Level column indicates no standard established for that compound.

U = compound was not detected
J = the reported value is an estimated cocentration
SA = Sample
DU = Duplicate Sample

FIGURES



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SENECA ARMY DEPOT ACTIVITY
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SEAD-16 AND SEAD-17

Figure 1
Location Map

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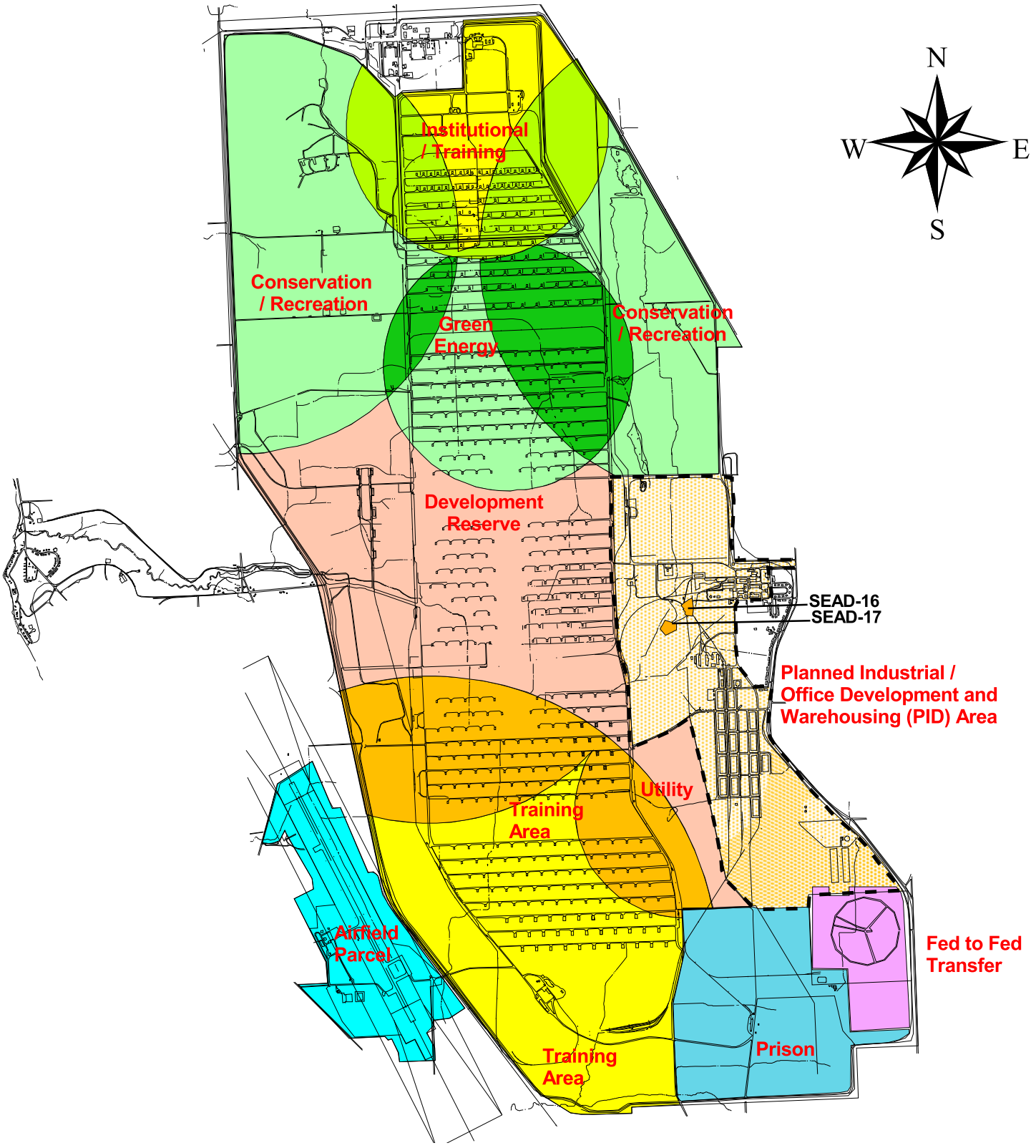

1000 0 1000 2000 3000 4000 5000 6000 7000 8000 9000 10000 11000 Feet

Area Covered by PID-wide Land Use Restrictions

- Prohibit the development and use of property for residential housing, elementary and secondary schools, childcare facilities and playgrounds.
- Prevent access to or use of the groundwater until the NYS Class GA Groundwater Standards are met.

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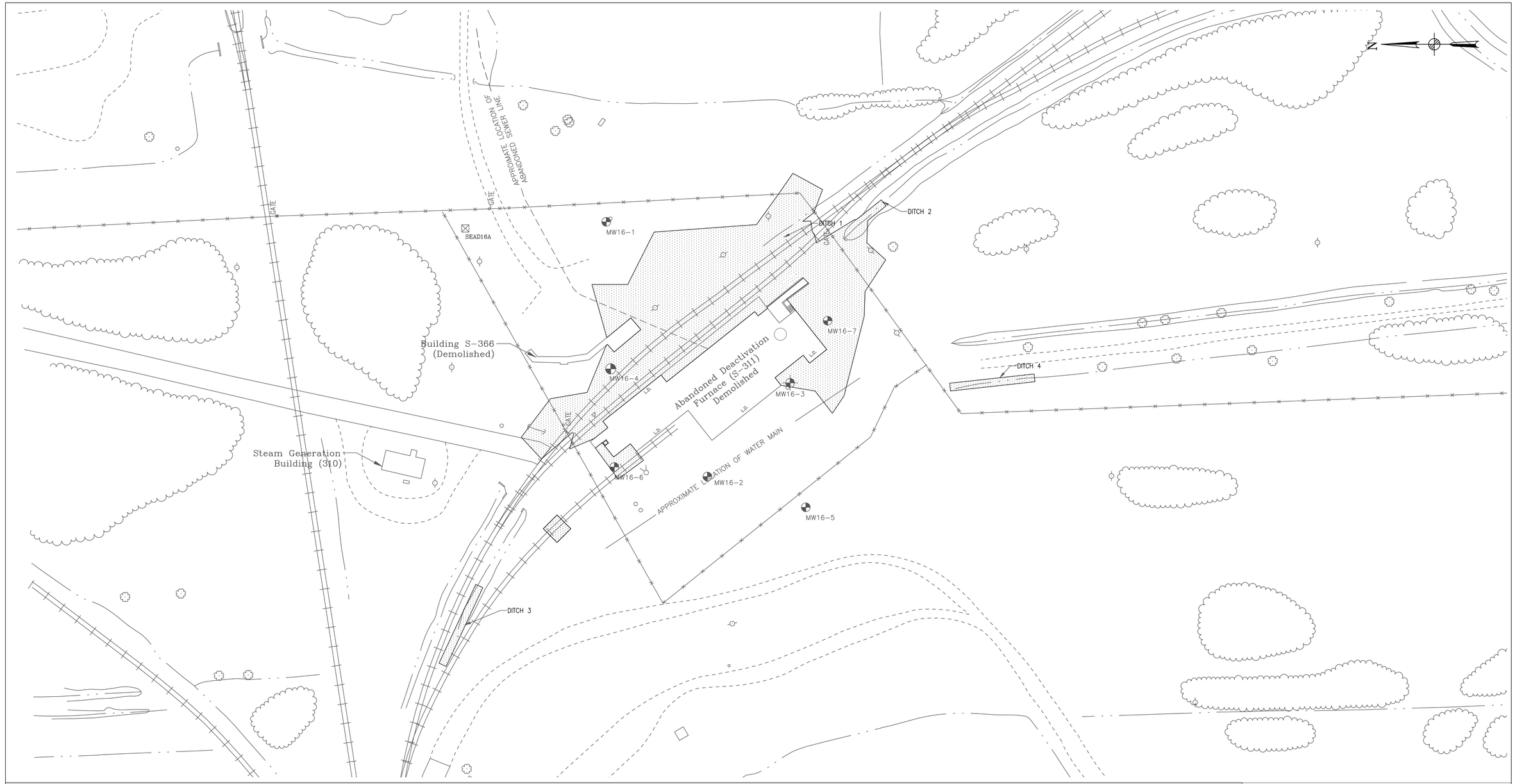
SENECA ARMY DEPOT ACTIVITY
ANNUAL REPORT - YEAR 7
FOR SEAD-16 AND SEAD-17

FIGURE 2
Location of SEAD-16 and SEAD-17
at Seneca Army Depot Activity

o:\seneca\pid luc\sead-16&17 annual report.apr\sead-16&17 Annual Report Figure 2



LEGEND:

- MINOR WATERWAY
- MAJOR WATERWAY
- FENCE
- BRUSH LINE
- RAILROAD
- UNPAVED ROAD
- SURVEY MONUMENT
- ROAD SIGN
- DECIDUOUS TREE
- L.D. LOADING DOCK
- FIRE HYDRANT
- MANHOLE
- GUIDE POST
- POLE
- UTILITY BOX
- OVERHEAD UTILITY POLE
- MAILBOX/RR SIGNAL
- MW16-5 MONITORING WELL LOCATION
- LIMITS OF EXCAVATION
- MW16-3 DESTROYED MONITORING WELL LOCATION

NOTE:

MONITORING WELL MW16-3 WAS DESTROYED DURING THE REMEDIAL ACTION.

50 0 50 100

SCALE IN FEET

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT ACTIVITY
ANNUAL REPORT – YEAR 8
SEAD-16 AND SEAD-17

DEPT. ENVIRONMENTAL ENGINEERING

Dwg. No. 748662-04500

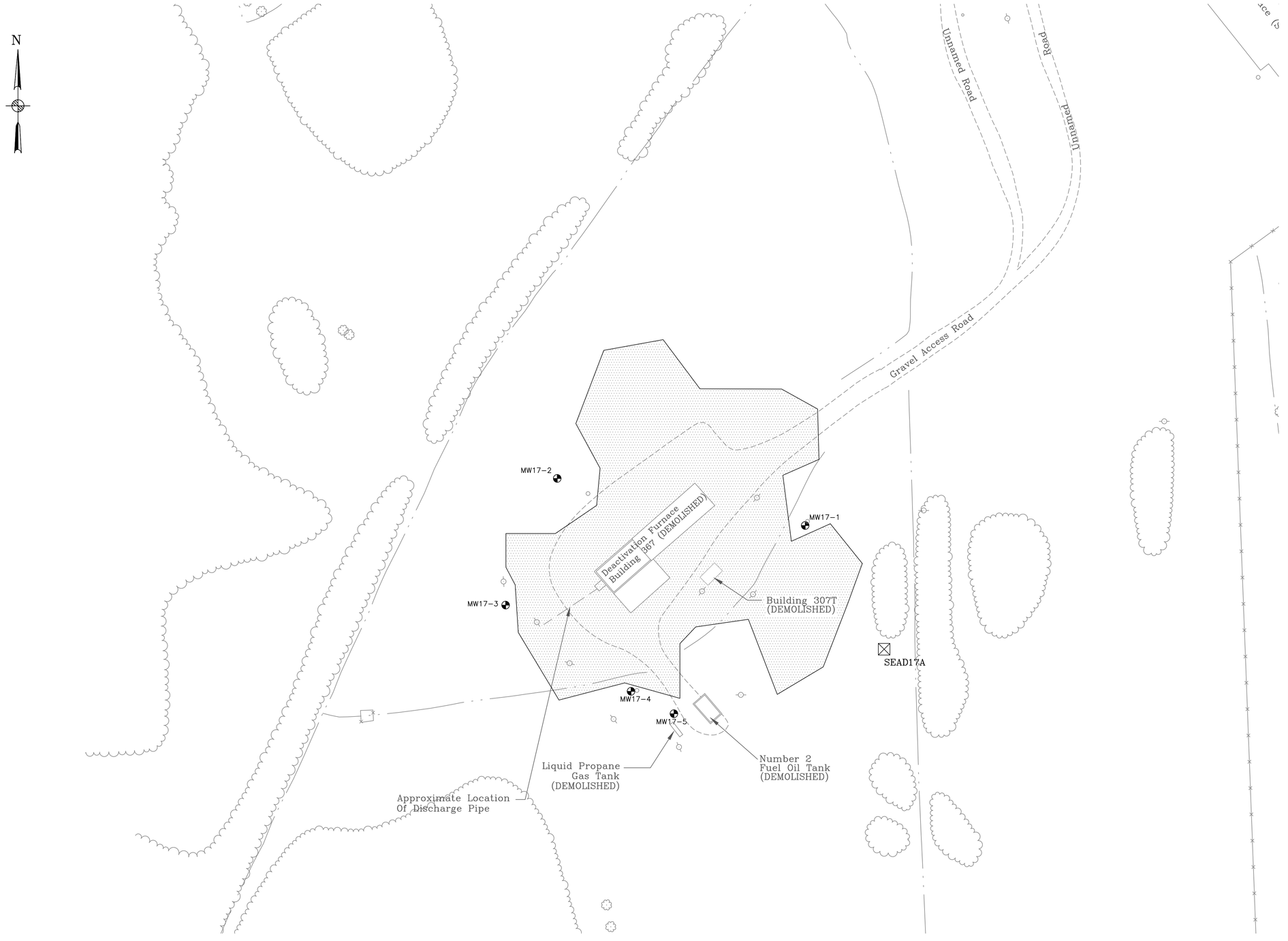
FIGURE 3
SEAD-16
SITE PLAN

SCALE 1" = 100'

DATE FEBRUARY 2016

REV –

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 – LTM AND LUC\SEAD 16 AND 17 LTM\ANNUAL REPORT – YR 8 DEC 2015\DRAFT\FIGURES\FIGURE 3.DWG, DATE: 03/16/2016 10:12:38AM, P0091241



LEGEND:

- MINOR WATERWAY
- MAJOR WATERWAY
- FENCE
- UNPAVED ROAD
- BRUSH LINE
- RAILROAD
- SURVEY MONUMENT
- ROAD SIGN
- DECIDOUS TREE
- GUIDE POST
- FIRE HYDRANT
- MANHOLE
- MAILBOX/RR SIGNAL
- POLE
- UTILITY BOX
- OVERHEAD UTILITY POLE
- MW17-5 MONITORING WELL LOCATION
- LIMITS OF EXCAVATION

PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT ACTIVITY
ANNUAL REPORT – YEAR 8
SEAD-16 AND SEAD-17

DEPT.	Dwg. No.
ENVIRONMENTAL ENGINEERING	748662-04500

FIGURE 4
SEAD-17
SITE PLAN

SCALE	DATE	REV
1" = 100'	FEBRUARY 2016	–

FILE: P:\PIT\PROJECTS\HUNTSVILLE CONT W912DY-08-D-0003\TO#15 - LTM AND LUC\SEAD 16 AND 17 LTM\ANNUAL REPORT - YR 8 DEC 2015\DRAFT\FIGURES\FIGURE 4.DWG, DATE: 03/16/2016 10:14:56AM, P0091241

LEGEND:

- 734 — ELEVATION CONTOUR
- MINOR WATERWAY
- MAJOR WATERWAY
- FENCE
- BRUSH LINE
- RAILROAD
- UNPAVED ROAD
- SURVEY MONUMENT
- ROAD SIGN
- DECIDUOUS TREE
- L.D. LOADING DOCK
- FIRE HYDRANT
- MANHOLE
- GUIDE POST
- POLE
- UTILITY BOX
- OVERHEAD UTILITY POLE
- MAILBOX/RR SIGNAL
- 729 — GROUNDWATER CONTOUR (DASHED WHERE INFERRED)
- MW17-3 — MONITORING WELL LOCATION
- LIMITS OF EXCAVATION
- MW17-3 (726.00) — APPROXIMATE GROUNDWATER ELEVATION
- INDICATES APPROXIMATE DIRECTION OF GROUNDWATER FLOW

NOTES:

1. MONITORING WELL MW16-3 WAS DESTROYED DURING THE REMEDIAL ACTION.
2. GROUNDWATER FLOW DIRECTION BASED ON DECEMBER 2014 GROUNDWATER DATA ELEVATION.

100 0 100 200
SCALE IN FEET

PARSONS

CLIENT/PROJECT TITLE
SENECA ARMY DEPOT ACTIVITY
ANNUAL REPORT – YEAR 8
SEAD-16 AND SEAD-17

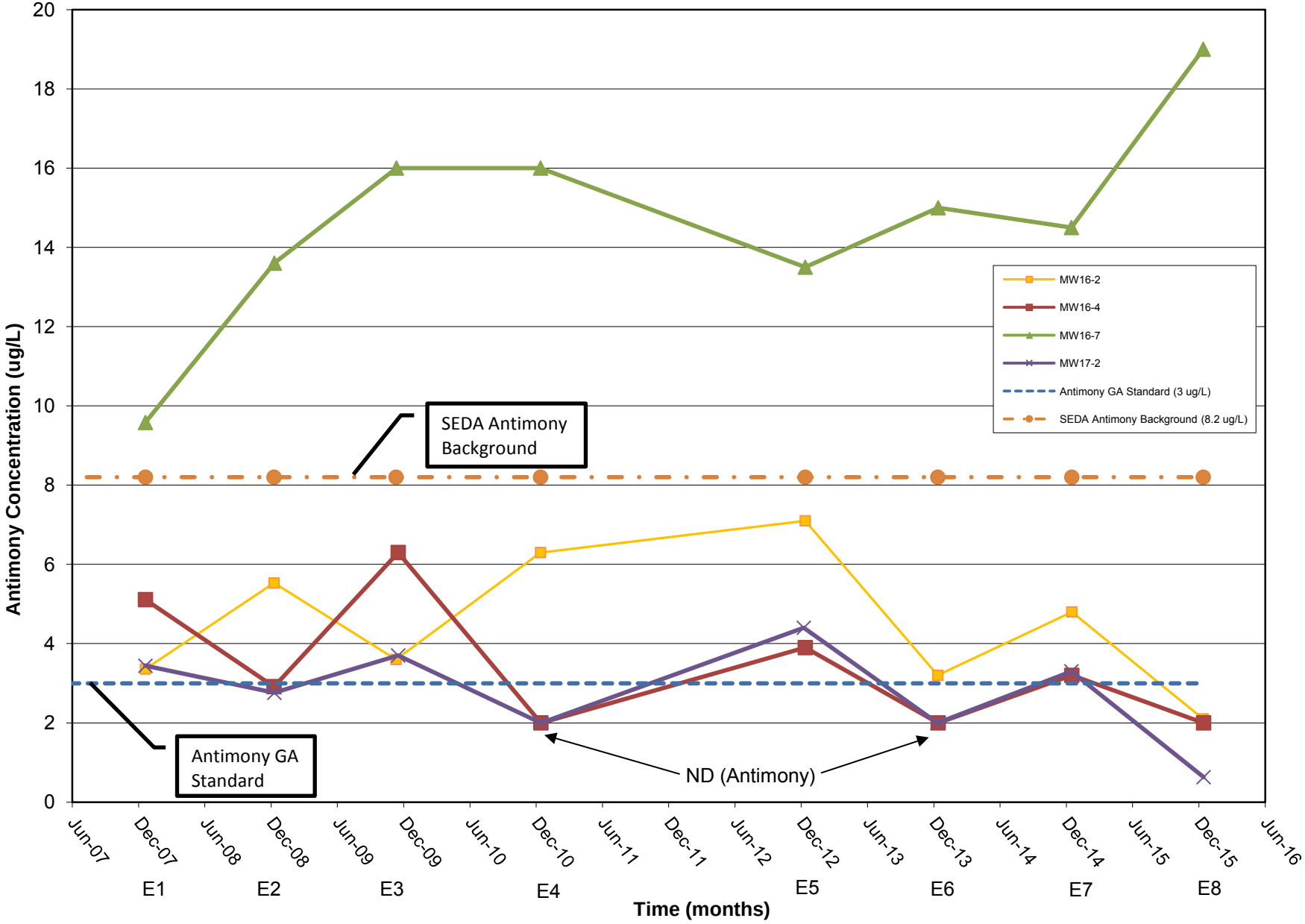
DEPT. ENVIRONMENTAL ENGINEERING
Dwg. No. 748662-04500

FIGURE 5
SEAD-16 AND SEAD-17
GROUNDWATER FLOW TREND

SCALE 1" = 200'
DATE FEBRUARY 2016
REV –

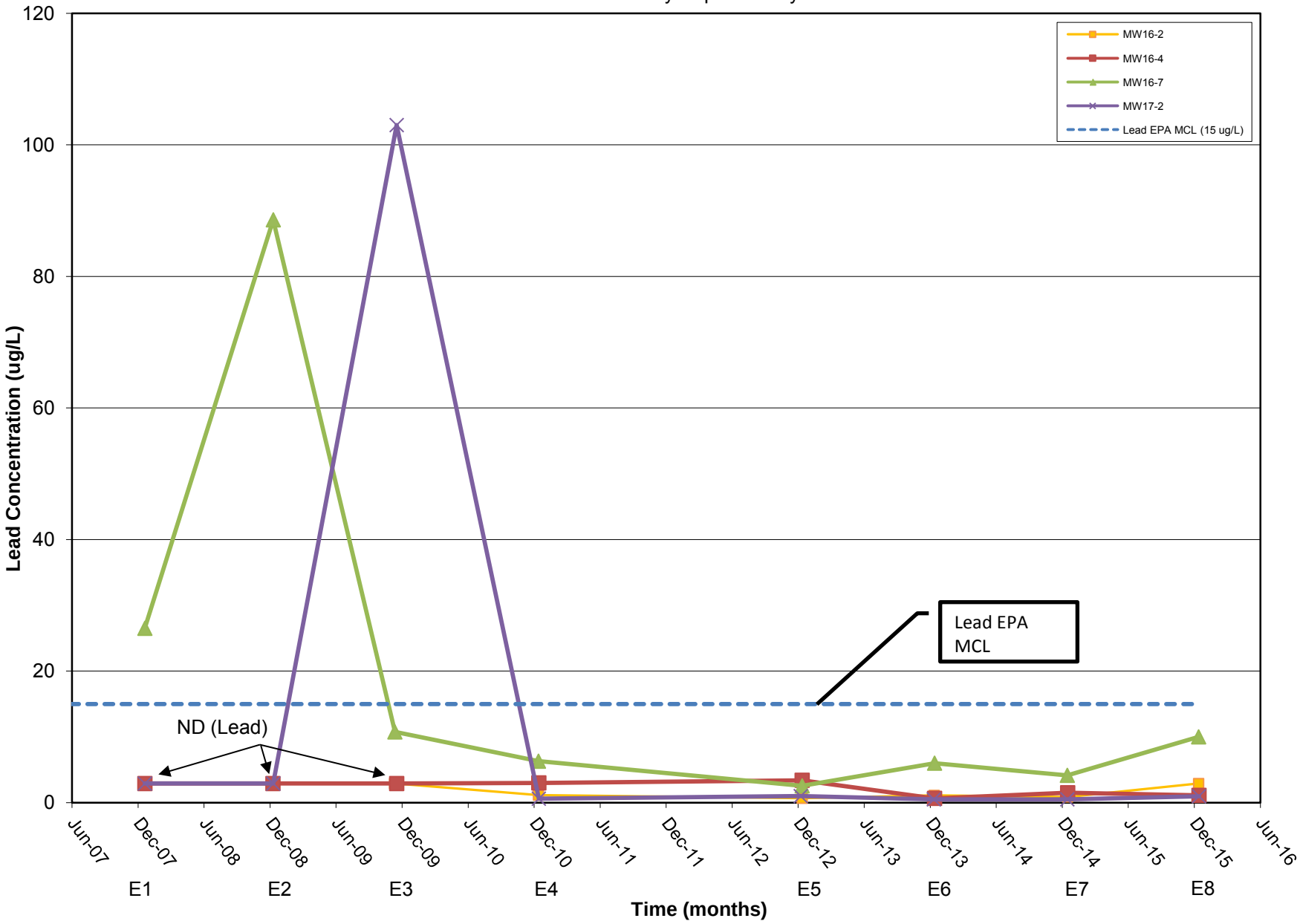
C:\USERS\P0091241\APPDATA\LOCAL\TEMP\ACPUBLISH_9248\FIGURE 5.DWG, DATE: 03/11/2016 02:17:56PM, P0091241

Figure 6A
Concentration of Antimony Over Time at MW16-2, MW16-4, MW16-7, and MW17-2
SEAD 16/17 Annual Report
Seneca Army Depot Activity



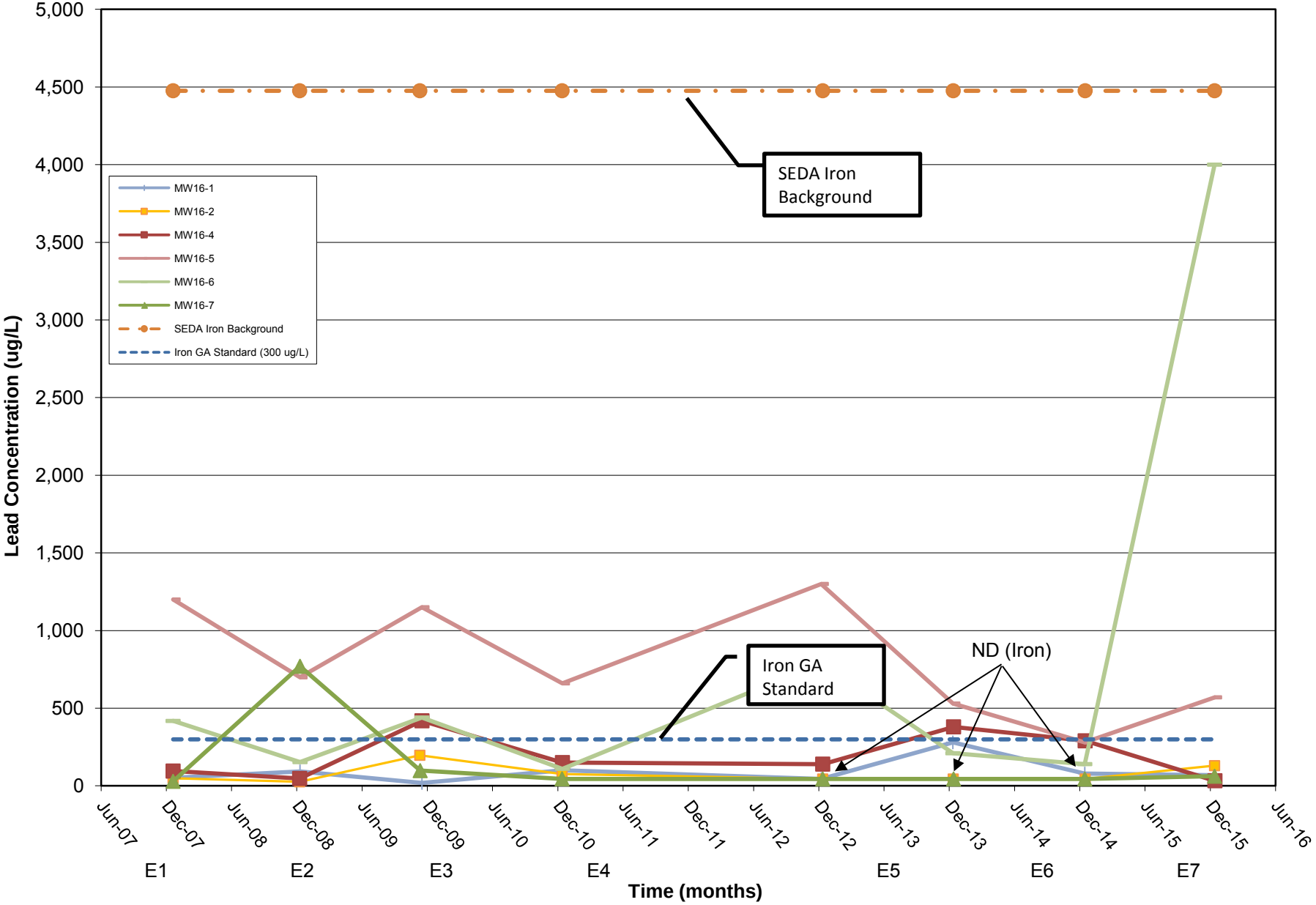
Note:
ND = not detected (MDL plotted).

Figure 6B
Concentration of Lead Over Time at MW16-2, MW16-4, MW16-7 and MW17-2
SEAD 16/17 Annual Report
Seneca Army Depot Activity



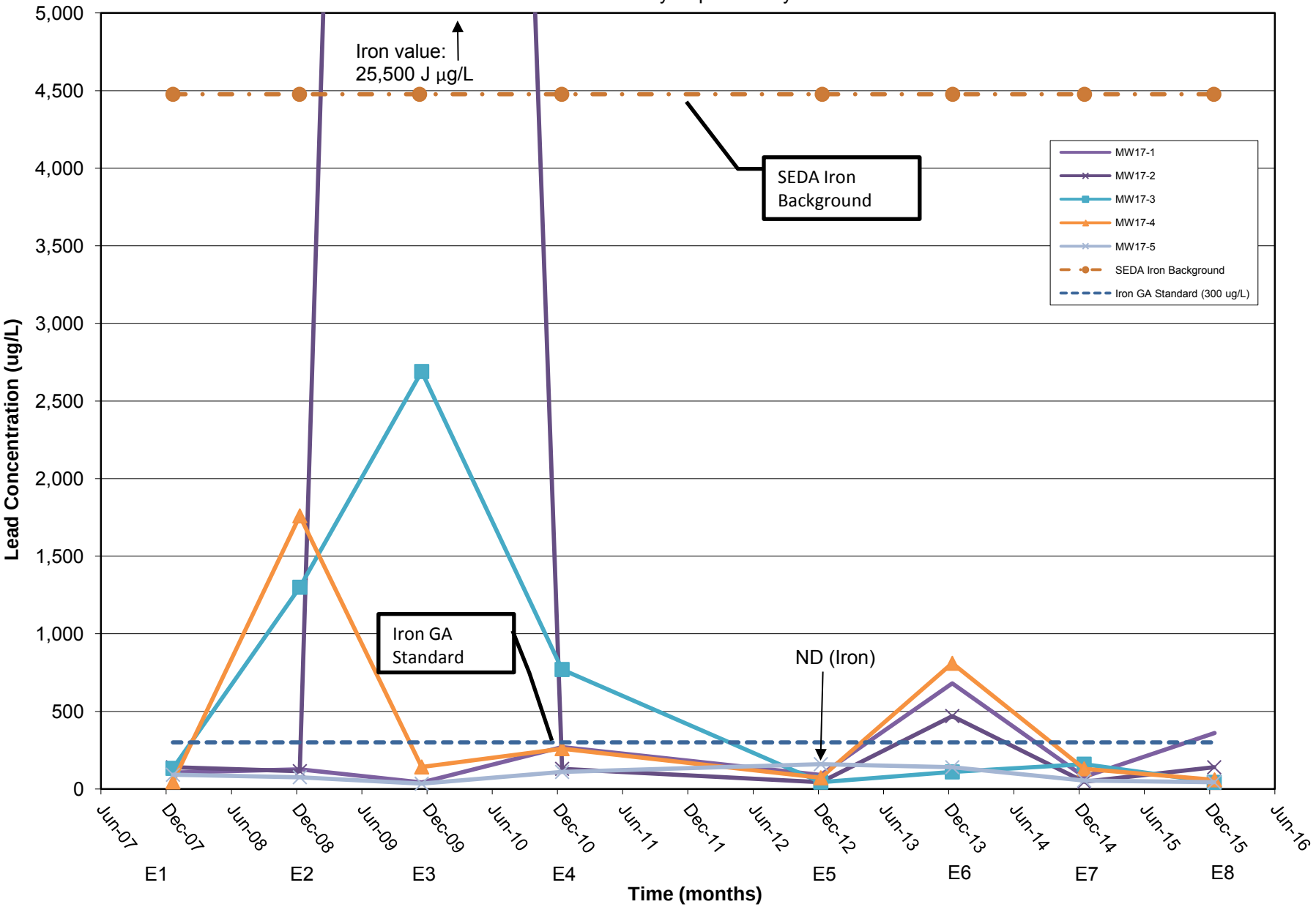
Note:
ND = not detected (MDL plotted).

Figure 6C
Concentration of Iron Over Time at SEAD 16 Monitoring Wells
SEAD 16/17 Annual Report
Seneca Army Depot Activity



Note:
ND = not detected (MDL plotted).

Figure 6D
Concentration of Iron Over Time at SEAD 17 Monitoring Wells
SEAD 16/17 Annual Report
Seneca Army Depot Activity



Note:
ND = not detected (MDL plotted).

APPENDICES

APPENDIX A

PRE-REMEDIAL ACTION MONITORING DATA

Appendix A
Pre-Remedial Action Groundwater Monitoring Results
Draft Annual Report - SEAD-16 and SEAD-17
Seneca Army Depot Activity

PARAMETER	ACTION LEVEL	SOURCE [1]	UNIT	LOC_ID:	MW16-1	MW16-1	MW16-2	MW16-2	MW16-3	MW16-3	MW16-4	MW16-4	MW16-5	MW16-6	MW16-6	MW16-7	MW16-7	MW16-7
				SAMP ID:	16101	16152	16102	16150	16110	16165	16105	16156	16162	16111	16155	16104	16158	16159
				QC CODE:	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA	DU
				STUDY ID:	RI ROUND1	RI ROUND2	RI ROUND1	RI ROUND2	RI ROUND1	RI ROUND2	RI ROUND1	RI ROUND2	RI ROUND2	RI ROUND1	RI ROUND2	RI ROUND1	RI ROUND2	RI ROUND2
				MATRIX:	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW	GW
				SAMPLE DATE:	8/27/1996	12/7/1996	8/27/1996	12/6/1996	8/30/1996	12/10/1996	8/28/1996	12/7/1996	12/9/1996	9/3/1996	12/8/1996	8/28/1996	12/8/1996	12/8/1996
SEMIVOLATILE ORGANICS																		
3-Nitroaniline	5	GA	UG/L		26 UJ	25 U	25 U	25 U	25 U	25 U	26 U	25 U	25 U	25 U	25 U	25 J	25 U	25 U
4-Chloroaniline	5	GA	UG/L		10 UJ	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 J	10 U	10 U
Benzo[ghi]perylene			UG/L		10 UJ	10 U	10 U	10 U	1 J	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
Dibenz[a,h]anthracene			UG/L		10 UJ	10 U	10 U	10 U	0.7 J	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
Diethyl phthalate			UG/L		10 UJ	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
Indeno[1,2,3-cd]pyrene			UG/L		10 UJ	10 U	10 U	10 U	0.6 J	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
OTHER ANALYSES																		
Nitrate/Nitrite Nitrogen	10	GA	MG/L		0.02	0.01 U	0.67	2	0.04	0.64	0.29	0.26	1.4	0.01 U	0.01 U	0.83	0.24	0.23
Percent Solids (Metals)					0	0	0	0	0	0	0	0	0	0	0	0	0	0
Total Petroleum Hydrocarbons			MG/L		0.44 U	0.4 U	0.4 U	0.36 U	0.41 U	1	0.41 U	0.42 U	0.91	0.89	0.73	0.41 U	0.46 U	1.3
NITROAROMATICS																		
1,3-Dinitrobenzene	5	GA	UG/L		0.26 U	0.26 U	1.8 J	0.26 U	0.26 U	0.26 U	0.26 U	0.26 U	0.26 U	0.26 U	0.26 U	0.26	0.26 U	0.26 U
2,4-Dinitrotoluene	5	GA	UG/L		0.26 U	0.26 U	0.26 U	0.26 U	0.26 U	0.26 U	0.68 J	0.26 U	0.26 U	0.26 U	0.26 U	0.26 U	0.26 U	0.26 U
METALS																		
Aluminum			UG/L		1850	143 U	1010	490	336	36.1 U	24.9	36.1 U	148 U	208	170 U	12.4	67.4 U	52.9 U
Antimony	3	GA	UG/L		2 U	3 U	2 U	3 U	7.5	5.3 U	2 U	3 U	3 U	2 U	3 U	15.7 U	8.9 U	10 U
Arsenic	10	MCL	UG/L		2.7 U	4.4 U	2.7 U	4.4 U	2.7 U	4.4 U	2.7 U	4.4 U	4.4 U	2.7 U	4.4 U	4 U	4.4 U	4.4 U
Barium	1,000	GA	UG/L		74.2	48.2 U	48.1	31.4 U	64.4	57.4 U	97.4	55.2 U	67.6 U	86.4	80.2 U	89.2	59.1 U	60.2 U
Beryllium	4	MCL	UG/L		0.23	0.2 U	0.22	0.2 U	0.21	0.2 U	0.21	0.2 U	0.2 U	0.1 U	0.2 U	0.21	0.2 U	0.2 U
Cadmium	5	GA	UG/L		0.3 U	0.6 U	0.3 U	0.6 U	0.3 U	0.6 U	0.3 U	0.6 U	0.6 U	0.3 U	0.6 U	0.3 U	0.6 U	0.6 U
Calcium			UG/L		157,000	116,000	193,000	164,000	99,800	85,500	130,000	158,000	90,000	44600	84,900	109,000	114,000	117,000
Chromium	50	GA	UG/L		2.7	1 U	2.3	1.1 U	1 U	1 U	1 U	1 U	1 U	1.5	1 U	1	1 U	1 U
Cobalt			UG/L		2.1	1.3 U	1.5	1.3 U	1.2 U	1.3 U	1.2 U	1.3 U	1.3 U	1.2	1.3 U	1.2	1.3 U	1.3 U
Copper	200	GA	UG/L		4.9	1.9 U	7.9	2.9 U	19.2	11.4 U	3.6	1.1 U	1.1 U	4.4	1.1 U	5.1	1.4 U	2.1 U
Iron	300	GA	UG/L		**2,400 J**	296	**1,720 J**	**923 J**	**432 J**	77.8 U	38.2	126	211	273 J	290	23.4	174	160
Lead	15	MCL	UG/L		1.7 U	1.5 U	5.9	6.8	6.1	1.5 U	1.7 U	1.5 U	3 U	1.7 U	1.5 U	8.4	9.9	9.2
Magnesium			UG/L		23,300	17,600	23,700	20,900	11,600	10,000	17,700	22,900	11,800	6370	12,800	16,900	22,600	23,200
Manganese	300	GA	UG/L		210	64.2	129	65.2	130	5.9 U	132	66.9	51	**545**	**1,380**	85.7	43.2	44.3
Mercury	0.7	GA	UG/L		0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U
Nickel	100	GA	UG/L		4.7	2.5 U	11	3.1 U	3	2.5 U	2.2	2.5 U	2.5 U	4.1	2.5 U	2.2	2.5 U	2.5 U
Potassium			UG/L		1670	998 U	4760	3410 U	2740	1900 U	4040	1660 U	18800	3530	2230 U	3220	2090 U	2160 U
Selenium	10	GA	UG/L		2.4 U	4.7 UJ	2.4 U	4.7 UJ	2.4 U	4.7 UJ	2.4 U	4.7 UJ	4.7 UJ	2.4 U	4.7 UJ	2.4 U	4.7 UJ	4.7 UJ
Sodium	20,000	GA	UG/L		8,750	3,870 U	19,100	17,000	9,480	7,660	17,200	12,300	**49,500**	**396000**	**409,000**	12,000	9,940	10,200
Thallium	2	MCL	UG/L		4.2 U	5.9 U	**9.2**	9.6 U	4.2 U	4.1 U	4.2 U	4.1 U	6.9 U	**6.2**	4.1 U	**4.2**	**11**	4.1 U
Vanadium			UG/L		3.3	1.6 U	2.9	1.6 U	1.2 U	1.6 U	1.2 U	1.6 U	1.6 U	2.9	1.6 U	1.2	1.6 U	1.6 U
Zinc			UG/L		15.6 R	5.8 U	37.4 R	13.5 U	32.4 R	42	4.5 R	5.1 U	6.3 U	13.2 R	10.5 U	2.9 R	2.2 U	7.3 U

Notes:
1. The criteria values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998) and EPA Maximum Contamination Limit (MCL), Source http://www.epa.gov/safewater/mcl.html#inorganic.html
2. Shading indicates a concentration above groundwater standard.
3. A blank in the action level column indicates no Class GA and/or MCL standand or standard is a secondary value.
4. Reported metals results are for total metals.

U = compound was not detected
J = the reported value is and estimated concentration
R = the compound was rejected
SA = Sample
DU = Duplicate

Appendix A
Pre-Remedial Action Groundwater Monitoring Results
Draft Annual Report - SEAD-16 and SEAD-17
Seneca Army Depot Activity

			LOC_ID:	MW17-1		MW17-1		MW17-1		MW17-2		MW17-3		MW17-4		MW17-5		MW17-5	
			SAMP ID:	16108		16109		16171		16163		16166		16169		16106		16170	
			QC CODE:	SA		DU		SA		SA		SA		SA		SA		SA	
			STUDY ID:	RI ROUND1		RI ROUND1		RI ROUND2		RI ROUND2		RI ROUND2		RI ROUND2		RI ROUND1		RI ROUND2	
			MATRIX:	GW		GW		GW		GW		GW		GW		GW		GW	
			SAMPLE DATE:	8/29/1996		8/29/1996		12/11/1996		12/9/1996		12/10/1996		12/11/1996		8/29/1996		12/11/1996	
PARAMETER	ACTION LEVEL	SOURCE [1]	UNIT	VALUE	Q	VALUE	Q	VALUE	Q	VALUE	Q	VALUE	Q	VALUE	Q	VALUE	Q	VALUE	Q
SEMIVOLATILE ORGANICS																			
Benzo[a]pyrene			UG/L	0.7	J	10	U	10	U	10	U	10	U	10	U	10	U	10	U
Benzo[ghi]perylene			UG/L	2	J	1	J	10	U	10	U	10	U	10	U	10	U	10	U
Dibenz[a,h]anthracene			UG/L	1	J	0.9	J	10	U	10	U	10	U	10	U	10	U	10	U
Indeno[1,2,3-cd]pyrene			UG/L	2	J	1	J	10	U	10	U	10	U	10	U	10	U	10	U
OTHER ANALYSES																			
Nitrate/Nitrite Nitrogen	10	GA	MG/L	0.24		0.23		0.2		0.04		0.05		0.02		0.04		0.02	
Percent Solids (Metals)				0		0		0		0		0		0		0		0	
NITROAROMATICS																			
Tetryl			UG/L	0.26	U	0.26	U	0.26	U	0.26	U	0.26	U	0.26	U	0.26	U	0.26	U
METALS																			
Aluminum			UG/L	90.4		54.6		386		85.3	U	36.1	U	41.9	U	39.9		59	U
Antimony	3	GA	UG/L	2	U	2	U	3	U	3	U	3	U	3	U	2	U	3	U
Arsenic	10	MCL	UG/L	2.7	U	2.7	U	4.4	U	4.4	U	4.4	U	4.4	U	2.7	U	4.4	U
Barium	1,000	GA	UG/L	85		87		90.4	U	66.1	U	27.4	U	27.4	U	92.5		62.6	U
Beryllium	4	MCL	UG/L	0.26		0.21		0.2	U	0.2	U	0.2	U	0.2	U	0.23		0.2	U
Cadmium	5	GA	UG/L	0.3	U	0.31		0.6	U	0.6	U	0.6	U	0.6	U	0.3	U	0.6	U
Calcium			UG/L	108000		110000		104000		118000		108000		92000		108000		81100	
Chromium	50	GA	UG/L	1	U	1.5		1	U	1	U	1	U	1	U	1	U	1	U
Cobalt			UG/L	1.2	U	1.4		2	U	1.3	U	1.3	U	1.3	U	1.2	U	1.3	U
Copper	200	GA	UG/L	3.1		4.3		1.1	U	2.6	U	1.1	U	1.1	U	3.3		1.3	U
Iron	300	GA	UG/L	119		90.6		**572**	**J**	214		53.1	U	96.4	U	56.8		134	
Lead	15	MCL	UG/L	1.7	U	1.7	U	1.5	U	1.9	U	1.5	U	3	U	1.7	U	1.5	U
Magnesium			UG/L	22600		23000		22900		14600		15200		14200		17700		13600	
Manganese	300	GA	UG/L	21.3		20		9.7	U	73.8		0.7	U	22.5		73.2		62	
Mercury	0.7	GA	UG/L	0.1	U	0.1	U	0.1	U	0.1	U	0.1	U	0.1	U	0.1	U	0.1	U
Nickel	100	GA	UG/L	1.8		2.2		2.5	U	2.5	U	2.5	U	2.5	U	2.4		2.5	U
Potassium			UG/L	472		574		843	U	5320		772	U	1330	U	853		1070	U
Selenium	10	GA	UG/L	2.4	U	2.4	U	4.7	UJ	4.7	UJ	4.7	UJ	4.7	UJ	2.4	U	4.7	UJ
Silver	50	GA	UG/L	1.3	U	2.3		1.5	U	1.5	U	1.5	U	1.5	U	1.3	U	1.5	U
Sodium	20,000	GA	UG/L	9,290		9,620		8,190		18,700		**30,100**		**22,300**		11,700		8,970	
Thallium	2	MCL	UG/L	**4.40**		**7.1**		4.1	U	4.7	U	4.4	U	6.2	U	**4.7**		8.6	U
Vanadium			UG/L	1.2	U	1.4		1.6	U	1.6	U	1.6	U	1.6	U	1.2	U	1.6	U
Zinc			UG/L	2.5	R	3.2	R	14.4	U	63.9		7.7	U	8.3	U	6.2	R	4.4	U

Notes:
1. The criteria values are NYSDEC Class GA Groundwater Standards (TOGS 1.1.1, June 1998) and EPA Maximum Contamination Limit (MCL), Source http://www.epa.gov/safewater/mcl.html#inorganic.html
2. Shading indicates a concentration above groundwater standard.
3. A blank in the action level column indicates no Class GA and/or MCL standand or standard is a secondary value.
4. Wells MW17-2, MW17-3, and MW17-4 were not sampled in August 1996 since they were dry.
5. Reported metals results are for total metals.

U = compound was not detected
J = the reported value is and estimated concentration
R = the compound was rejected
SA = Sample
DU = Duplicate

APPENDIX B

SEDA BACKGROUND GROUNDWATER DATA SUMMARY

Appendix B
SEDA Background Groundwater Concentrations
Draft Annual Report - Year 8 for SEAD-16 and SEAD-17
Seneca Army Depot Activity

PARAMETER	UNIT	MAXIMUM	AVERAGE CONCENTRATION	STANDARD DEVIATION	FREQUENCY OF DETECTION	CRITERIA VALUE	TYPE OF CRITERIA	NUMBER OF EXCEEDENCES	NUMBER OF DETECTS	NUMBER OF ANALYSES
Aluminum	UG/L	42,400	2,732	8,207	87%	50	MCL	25	27	31
Antimony	UG/L	52.7	8.2	13.9	13%	3	GA	3	4	31
Arsenic	UG/L	10	1.7	2.2	13%	10	MCL	2	4	31
Barium	UG/L	337	78.2	62.6	94%	1000	GA	0	29	31
Beryllium	UG/L	2.2	0.2	0.4	13%	4	MCL	0	4	31
Cadmium	UG/L	0	0.5	0.5	0%	5	GA	0	0	31
Calcium	UG/L	181,000	115,619	25,274	100%			0	31	31
Chromium	UG/L	69.4	4.7	13.4	48%	50	GA	1	15	31
Cobalt	UG/L	34.6	3.7	7.4	45%			0	14	31
Copper	UG/L	32.5	3.3	6.9	48%	200	GA	0	15	31
Cyanide	UG/L	2.8	NA	NA	3%	200	GA	0	1	31
Iron	UG/L	69,400	4,476	13,429	100%	300	GA	22	31	31
Lead	UG/L	34.8	2.5	6.3	32%	15	MCL	1	10	31
Magnesium	UG/L	58,200	28,568	13,848	100%			0	31	31
Manganese	UG/L	1120	224	254	97%	300	SEC	22	30	31
Mercury	UG/L	0.06	0.04	0.02	23%	0.7	GA	0	7	31
Nickel	UG/L	99.8	7.3	18.7	61%	100	GA	0	19	31
Potassium	UG/L	10,200	3,833	3,010	94%			0	29	31
Selenium	UG/L	3.6	1.5	0.7	19%	10	GA	0	6	31
Silver	UG/L	0.98	1.0	1.0	6%	50	GA	0	2	31
Sodium	UG/L	59,400	14,601	13,877	97%	20000	GA	7	30	31
Thallium	UG/L	4.7	1.5	1.2	13%	2	MCL	4	4	31
Vanadium	UG/L	70.8	5.2	13.5	52%			0	16	31
Zinc	UG/L	143	23.1	34.5	84%	5000	MCL	0	26	31

GA = NYSDEC Ambient Water Quality Standards for a source of Drinking Water from Groundwater (TOGS 1.1.1)
MCL = Maximum Contaminant Level - Drinking Water Standards and Health Advisory (EPA 822-B-00-001)
SEC = Secondary Drinking Water Regulations - Drinking Water Standards and Health Advisory (EPA 822-B-00-001)

APPENDIX C

FIELD FORMS - YEAR 8 LTM GROUNDWATER SAMPLING ACTIVITIES

GROUNDWATER ELEVATION REPORT

PARSONS | CLIENT: | DATE: 12/16/15

PROJECT: S-16/17 | PROJECT NO:

LOCATION: | INSPECTOR: BBO / DD

MONITORING EQUIPMENT:

INSTRUMENT	DECTECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR:

INSTRUMENT	CORRECTION FACTOR

COMMENTS: overcast, winds 5-10 SW→NE

WELL	TIME	DEPTH TO WATER	DEPTH TO PRODUCT	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC. GRAV.	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of: riser, concrete, protective casing, etc.)
17-2	1450	3.80	6.10					locked,
17-3	1453	4.40	7.49					? unlocked
17-4	1456	3.40	8.20					locked, no well cap
17-5	1458	3.09	10.18					locked
17-1	1503	3.59	9.95					locked, mouse nest removed
16-5	1511	2.02	5.09					PVC lifted, well cap crushed into PVC top, locked
16-7	1513	3.60	6.78					locked
16-1	1516	3.93	7.95					locked, PVC recessed in metal well case
16-6	1521	2.80	6.85					locked
16-4	1523	3.18	7.08					locked
16-2								unable to open lock

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

GROUNDWATER ELEVATION REPORT

PARSONS | CLIENT: | DATE: 12/19/15

PROJECT: SEAD-16/17 LTM Round 10 | PROJECT NO:

LOCATION: | INSPECTOR: TBB0 / DD

COMMENTS: overcast, 4t show shower

MONITORING EQUIPMENT:

INSTRUMENT	DECTECTOR	BGD	TIME	REMARKS

WATER LEVEL INDICATOR:

INSTRUMENT	CORRECTION FACTOR

WELL	TIME	DEPTH TO WATER	DEPTH TO ~~PRODUCT~~ Depth	CORRECTED WATER LEVEL	MEASURED POW	INSTALLED POW	PRODUCT SPEC. GRAV.	WELL STATUS / COMMENTS (Lock?, Well #?, Surface Disturbance?, Riser marked?, Condition of riser, concrete, protective casing, etc.)
17-1	1139	3.55	9.95					Mouse nest rebuilt
17-5	1142	2.96	10.18					
17-4	1145	3.28	8.20					
17-3	1147	3.73	7.49					
17-2	1149	~~3.45~~ → 3.56		6.10				
16-2	1155	3.33	5.65					
16-5	1157	2.20	5.09					
16-7	1158	3.52	6.78					
16-1	1200	3.69	7.95					
16-4	1202	3.03	7.08					
16-6	1203	2.68	6.85					
		* See 12/16/15 GW survey form for well condition comments.						

(ALL DEPTH MEASUREMENTS FROM MARKED LOCATION ON RISER)

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW 16-2

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12/12/15
INSPECTORS: BBO
PUMP #: Parsons Peristaltic
SAMPLE ID #: 16LM20050

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND DIRECTION (FROM) (0 - 360)	GROUND / SITE SURFACE CONDITIONS
1212	30s	overcast		10-20	SW-NE	

MONITORING INSTRUMENT	MONITORING DETECTOR

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	5.65					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		3.33'			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
1222	3.31	YSI	Inwell	YSI	YSI	Horiba	Horiba	Horiba	LaMotte
1222		Peristaltic pump Started							
1232	3.34	114		3.75	8.5	0.399	7.52	93	33.8
1237	3.34			1.56	8.4	0.444	7.40	103	20.1
1242	3.34			0.98	8.5	0.485	7.35	107	9.85
1247	3.34	~120	~0.5 gals	1.60	8.6	0.476	7.32	101	87.7
1252	"			0.76	8.6	0.526	7.29	91	25.1
1257	"		~1.0 gal	0.61	8.7	0.554	7.25	80	10.31
1302	"			0.50	8.7	0.565	7.22	70	4.79
1307	"		~1.25 gals	0.40	8.6	0.573	7.21	63	3.06
1312	"		~1.5 gals	0.34	8.7	0.578	7.21	58	2.40
1317	3.34		~1.6 gals	0.31	8.7	0.583	7.22	57	2.44
1320		Sample	Collected	1x Plastic	for	Metals	unfiltered		
1323		Restarted	Pump to Collect Post-Sample Collected for Pars						
1328	3.34		~1.9 gals	0.20	8.7	0.592	7.21	57	7.60

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW16-5

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12/19/15
INSPECTORS: DRD
PUMP #: Peristaltic
SAMPLE ID #: 16LM20052

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS
1215	20s	overcast, windy		10-20	55W	Gravel

MONITORING INSTRUMENT	MONITORING DETECTOR

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	5.09					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		2.17			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
1220	2.17	YSI	Installed	YSI	YSI	Horiba	Horiba	Horiba	LaMotte
1225		120		0.55	6.9	0.446	6.97	233	12.20
1230				0.38	7.2	0.443	6.63	134	
1235		120		0.33	7.3	0.440	6.81	77	
1240	3.45			0.30	7.4	0.440	6.81	22	3.90
1245				0.26	7.6	0.441	6.83	-5	
1250				0.24	7.7	0.443	6.77	-28	
1255				0.22	7.8	0.445	6.76	-40	
1300	3.85	120		0.18	7.9	0.447	6.77	-55	1.53
1305				0.16	8.0	0.449	6.72	-62	
1310	4.15	120	~ 2.0 gals	0.14	8.2	0.452	6.69	-74	1.94
1315	Collected		Sample # 16LM20052						
~~1320~~	Collecting		post sampling	round of	Geo	parameters			
1320		120		0.11	8.1	0.453	6.75	-81	0.94

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW16-7

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12/19/15
INSPECTORS: DRD
PUMP #: Peristaltic
SAMPLE ID #: 16LM20054 / MS/MSD Duplicate - 16LM20055

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND (FROM) VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS
1345	20s	overcast, windy		10-20	WSW	Gravel

MONITORING INSTRUMENT	MONITORING DETECTOR

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	6.78					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		3.48			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
1345	3.48	YSI	Installed	YSI	YSI	Horiba	Horiba	Horiba	LaMotte
1350		140		3.23	9.1	0.485	7.14	-67	43.2
1355				3.01	9.3	0.473	7.05	-56	
1400	4.00			3.09	9.4	0.480	6.96	-35	
1405		160		2.78	9.3	0.485	6.98	-30	5.36
1410				2.07	9.4	0.492	6.94	-21	
1415		152		1.62	9.6	0.499	6.91	-15	
1420	4.02			1.44	9.7	0.512	6.94	-11	
1425		155		1.35	9.8	0.524	6.91	-5	2.97
1430				1.30	9.8	0.534	6.94	-3	
1435	4.03			1.27	9.8	0.544	6.92	1	1.62
1440	Collected		Sample #5	16LM20054					
				16LM20054 MS					
				16LM20055 (Duplicate)			Time =	1500	
	Collected		post sample	Geo parameter readings					
1445			~2.5 gals	1.17	9.8	0.595	6.90	14	1.92

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW16-6

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12/19/15
INSPECTORS: BBO
PUMP #: Parsan Peristaltic
SAMPLE ID #: 16LM20053

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND DIRECTION (FROM) (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
1400	30's	overcast		10-20	SW→NE			

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
1 x well Vol = 0.68 gals 3 x Well = 2.0 gals

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	6.85'					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		2.68'			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
1410 ~~2.10~~	2.59	YSI	2 tubing in the well	YSI	YSI	Horiba	Horiba	Horiba	LaMotte
1410		Peristaltic Pump Started							
1414	3.34	94		2.10	9.1	0.387	~~7.43~~	105	7.67
1419	3.72	102		2.14	9.1	0.385	7.40	108	6.21
1424	3.95			2.03	9.1	0.386	7.36	103	4.18
1429	4.1			2.10	9.2	0.386	7.34	98	3.86
1434	4.24			1.84	9.2	0.380	7.33	88	2.75
1439	4.35		0.5 gals	1.66	9.3	0.391	7.32	68	2.03
1444	4.45	118		1.65	9.3	0.391	7.33	51	1.39
1449	4.54			1.75	9.3	0.380	7.34	41	1.18
1454	4.63		1.0 gals	1.71	9.3	0.384	7.34	36	1.13
1459	4.73			1.71	9.3	0.381	7.35	33	0.87
1504	4.83	122	~1.25 gals	1.76	9.3	0.379	7.36	26	0.91
1509	4.95			1.68	9.3	0.384	7.35	11	1.09
1514	5.05		~1.5 gal	1.21	9.4	0.391	7.33	-13	1.34
1519	5.15	110		1.37	9.4	0.398	7.32	-27	1.39
1524	5.23		~2.0 gals	1.62	9.5	0.400	7.31	-33	1.86
1530		Sample Collected		1x Plastic for Metals			Unfiltered		
1531		Post-Sample Gen Purge Pump Started							
1536	5.43		2.25	1.49	9.6	0.405	7.29	-30	5.73

5.43

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW16-4

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12-20-15
INSPECTORS: DRS
PUMP #: Peristaltic
SAMPLE ID #: 16LM20051

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
0755	30s	P/cloudy		5-10	WNW	Grass		

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
Well Vol = 0.632 gals

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	7.08					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		3.13			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS L. Motte

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L) YSI	TEMP (C)	SPEC. COND (umhos) Horiba	pH	ORP (mV)	TURBIDITY (NTU)
0800	3.08	YSI	Installed	0.81	8.6	1.32	6.89	239	
0805	3.25	150		0.69	8.6	1.35	6.91	237	3.55
0810				0.49	8.5	1.43	6.88	236	
0815	3.26	160		0.38	8.5	1.56	6.84	235	1.27
0820				0.35	8.4	1.63	6.82	234	
0825	3.25	160		0.27	8.4	1.69	6.81	233	
0830				0.26	8.4	1.74	6.80	232	
0835	3.26	165	~ 2.5 gals	0.27	8.4	1.75	6.79	231	1.19
0840		Collected Sample #		16LM20051					
0845		Collecting post sample		Geo parameter readings					
0845	3.26			0.22	8.4	1.82	6.82	233	0.87

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW16-1

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12/20/15
INSPECTORS: BBO
PUMP #: Rossa Peristaltic
SAMPLE ID #: 16LM20049

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
742	30's	partly cloudy		0-5	S-2N	grassy		

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
1x Well Vol = 0.66 gals 3x = 1.98 gals

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	7.95'					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
0.78' diff between TOR & TOC		3.91			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

TOR TOC

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
3.89									
809	4.71	YSI ?	tubing in well	YSI	YSI	Horiba	Horiba	Horiba	LaMotte
814		Pump	Started						
819	4.79	106		1.40	9.5	0.808	7.42	213	7.98
824	4.79			1.65	9.6	0.816	7.37	218	15.5
829	"	~118		1.30	9.6	0.822	7.29	208	8.04
834	4.8			1.11	9.6	0.824	7.24	206	4.98
839	4.8	126	~0.5 gals	0.98	9.6	0.826	7.16	213	3.38
844	4.8			0.79	9.6	0.826	7.12	212	2.78
849	4.82		~0.75 gal	1.01	9.7	0.830	7.08	200	2.53
854	4.82	132	~1.0 gal	0.85	9.7	0.832	7.07	189	2.34
859	4.8			0.65	9.7	0.832	7.04	181	2.11
904	4.8		~1.5 gals	0.48	9.7	0.834	7.02	167	1.76
909	4.81	134		0.37	9.7	0.836	7.00	146	1.60
914	4.81		~1.75 gals	0.30	9.7	0.836	7.00	134	1.34
919	4.81			0.33	9.7	0.836	7.00	124	1.09
924	4.81		~2.0 gals	0.34	9.7	0.836	6.99	107	1.02
927		Sample	Collected	1x Plastic	for	Metals	unFiltered		
929	4.79	Pump restarted to collect - Post-Sample				Collect	Geo Para		
934	4.81		~2.5 gals	0.32	9.7	0.838	6.98	99	0.92

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW17-2

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12-20-15
INSPECTORS: DRS
PUMP #: Peristaltic
SAMPLE ID #: 17LM 20036

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
0930	30s	Clear		0-5	WNW	Grass/brush		

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
Well Vol = 0.392

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	6.10'					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		3.65			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
0930	3.58	YSI	Probe installed YSI		YSI	Horiba	Horiba	Horiba	LaMotte
0935		180		1.27	8.4	0.817	7.62	192	
0940	4.65	160		0.95	8.7	0.789	7.24	181	11.25
0945	4.95	112		0.82	8.8	0.774	7.09	135	3.20
0950	5.45			0.75	8.8	0.764	6.96	73	
0955		120		0.49	8.9	0.761	6.90	41	
1000	5.80	112		0.36	8.9	0.758	6.88	26	1.38
1005	5.88	108		0.29	9.0	0.755	6.88	14	
1010			~ 1.5 gals	0.27	9.0	0.756	6.86	6	1.41
1015	Collected		Sample #	17LM 20036	-	Well very close to going dry		>	3 vols removed
	Collected		post sampling	Geo parameters					
1020	5.93			0.27	9.0	0.750	6.98	-3	0.79

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW17-5

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12/20/15
INSPECTORS: TSTO
PUMP #: Parsons Peristaltic
SAMPLE ID #: 17LM20039

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
949	30's	sunny		1-5	SO-SW	grassy		

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
1x well = 1.16 gals 3x = 3.49 gals

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	10.18'					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		3.05'			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
959	3.05	YSI &	tubing on well	YSI	YSI	Horiba	Horiba	Horiba	LaMotte
1000		Pump	Started						
1004	3.15	126		0.53	9.7	0.543	7.40	165	3.28
1009	3.16			0.44	9.9	0.539	7.22	185	3.42
1014	3.18	158		0.34	9.9	0.536	7.20	193	1.31
1019	3.19			0.30	10.0	0.535	7.18	168	1.28
1024	3.18		~0.75 gals	0.30	9.7	0.529	7.19	141	0.90
1029	3.18			0.28	9.8	0.527	7.19	128	0.92
1034	3.17	142	~1.0 gals	0.24	9.7	0.525	7.20	109	1.38
1039	3.16			0.25	9.8	0.525	7.22	88	0.75
1044	3.15		~1.5 gals	0.25	9.8	0.525	7.23	84	1.06
1049	3.17	126		0.21	10.0	0.524	7.25	76	0.64
1054	3.16		~2.0 gals	0.19	10.1	0.525	7.26	68	0.81
1059	3.15			0.19	10.1	0.523	7.26	63	0.63
1104	3.16		~2.3 gals	0.18	10.1	0.521	7.27	57	0.67
1109	3.16			0.17	10.1	0.521	7.28	52	0.63
1114	3.16	148	~2.6 gals	0.19	10.1	0.520	7.28	45	0.98
1119	3.15			0.18	10.1	0.519	7.28	42	0.71
1124	3.16	122	~3.0 gals	0.12	10.1	0.519	7.28	40	0.70
1129	3.17		~3.2 gals	0.09	10.6	0.519	7.28	39	0.69
1134	3.16			0.08	10.1	0.519	7.28	36	1.37

1137 Sample Collected
1139 Restarted Pump for Post-Sample Collection Geo Parameters

1144	3.15		~3.75 gals	0.08	10.1	0.519	7.27	36	0.59

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW17-3

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12-20-15
INSPECTORS: DRJ
PUMP #: Peristaltic
SAMPLE ID #: 17LM20037

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	(FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
1045	30s	Clear		0-5	NNW	Grass/Gravel		

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
Well Vol. = 0.584

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	7.49					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		3.84			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
1050	~~7.35~~ DRY	3.75	YSI Probe Installed	YSI	YSI	Horiba	Horiba	Horiba	LaMotte
1055				5.18	9.1	0.514	7.17	27	
1100	4.97	120		4.86	9.0	0.508	7.19	33	47.1
1105	5.18	108		4.89	8.9	0.504	7.00	54	
1110	5.35			4.56	9.0	0.501	6.94	65	5.25
1115	5.44	112		4.14	9.0	0.502	6.86	77	
1120				4.30	9.1	0.503	6.84	82	
1125	5.56	110		4.05	9.1	0.504	6.79	88	
1130				3.54	9.2	0.504	6.78	95	3.58
1135	5.70	116		3.50	9.2	0.504	6.81	95	
1140				3.22	9.2	0.505	6.78	98	
1145	5.82	112		2.95	9.3	0.506	6.82	98	0.89
1150				2.58	9.3	0.505	6.78	103	
1155	5.93			2.30	9.3	0.506	6.75	106	
1200		118		2.06	9.3	0.506	6.77	106	
1205	6.06		~2.0 gals	1.81	9.3	0.507	6.76	108	1.76
1210	Collected		Sample # 17LM20037		-	> 3 well vols removed			
	Collected		post sample	Geo parameters					
1215				1.83	9.4	0.536	6.82	107	1.87

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW17-1

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12/21/15
INSPECTORS: DRD
PUMP #: Peristaltic
SAMPLE ID #: 17LM20035

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND (FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS
0745	40s	Overcast		5-10	SSW	Grass/Brush

MONITORING	
INSTRUMENT	DETECTOR

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
Well Vol. = 1.01 gals

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	9.95'					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		3.62			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
0750	3.54		YSI Probe installed						
0755				0.17	10.4	0.438	7.72	207	
0800	4.10	150		0.13	10.5	0.430	7.46	181	9.47
0805	4.16			0.09	10.5	0.441	7.35	61	
0810		160		0.08	10.5	0.439	7.35	17	
0815	4.23			0.07	10.6	0.459	7.31	-16	4.99
0820		160		0.06	10.6	0.480	7.28	-30	
0825	4.25			0.06	10.6	0.489	7.25	-35	3.45
0830				0.06	10.6	0.500	7.23	-39	
0835	4.29			0.05	10.6	0.512	7.22	-42	
0840		164		0.05	10.6	0.523	7.22	-44	2.06
0845				0.05	10.7	0.514	7.22	-46	
0850		170	~ 2.5 gals	0.05	10.7	0.524	7.23	-47	1.36
0855	Collected		Sample # 17LM20035						
	Collected		Post Sample Geo parameters						
0900	4.30			0.04	10.7	0.534	7.26	-51	1.61

SAMPLING RECORD - GROUNDWATER

SENECA ARMY DEPOT ACTIVITY	PARSONS	WELL #: MW 17-4

PROJECT: SEAD-16/17 LTM Groundwater Sampling - Round 8
LOCATION: ROMULUS, NY

DATE: 12/21/15
INSPECTORS: BJB O
PUMP #: Parsons Peristaltic
SAMPLE ID #: 17LM20038

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

TIME (24 HR)	TEMP (APPRX)	WEATHER (APPRX)	REL. HUMIDITY (GEN)	WIND VELOCITY (APPRX)	WIND DIRECTION (FROM) (0 - 360)	GROUND / SITE SURFACE CONDITIONS	MONITORING INSTRUMENT	MONITORING DETECTOR
745	30s	overcast		5-10	SW→NE	grassy		

WELL VOLUME CALCULATION FACTORS

DIAMETER (INCHES):	0.25	1	2	3	4	6
GALLONS / FOOT:	0.0026	0.041	0.163	0.367	0.654	1.47
LITERS/FOOT	0.010	0.151	0.617	1.389	2.475	5.564

ONE WELL VOLUME (GAL) = [(POW - STABILIZED WATER LEVEL) X WELL DIAMETER FACTOR (GAL/FT)]
1x Well Vol = 0.79 3x = 2.38 gal

HISTORIC DATA	DEPTH TO POINT OF WELL (TOC)	DEPTH TO TOP OF SCREEN (TOC)	SCREEN LENGTH (FT)	WELL DEVELOPMENT TURBIDITY	WELL DEVELOPMENT pH	WELL DEVELOPMENT SPEC. COND
	8.20'					

DATA COLLECTED AT WELL SITE	PID READING (OPENING WELL)	DEPTH TO STATIC WATER LEVEL (TOC)	DEPTH TO STABILIZED WATER LEVEL (TOC)	DEPTH TO PUMP INTAKE (TOC)	PUMPING START TIME
		3.33'			

RADIATION SCREENING DATA	PUMP PRIOR TO SAMPLING (cps)		PUMP AFTER SAMPLING (cps)	

MONITORING DATA COLLECTED DURING PURGING OPERATIONS

TIME (min)	WATER LEVEL	PUMPING RATE (ml/min)	CUMULATIVE VOL (GALLONS)	DISSOLVED OXYGEN (mg/L)	TEMP (C)	SPEC. COND (umhos)	pH	ORP (mV)	TURBIDITY (NTU)
752	3.30	YSI?	tubing mod						
752		Pump Started							
800	3.62	100		2.36	8.0	0.435	7.63	205	3.09
805	3.69	114		1.36	8.2	0.434	7.55	211	2.74
810	3.79			1.41	8.2	0.437	7.54	211	2.28
815	3.84	110		1.25	8.2	0.441	7.49	210	2.39
820	3.87		~0.5 gal	0.64	8.2	0.442	7.43	198	2.18
825	3.93			0.56	8.1	0.445	7.35	160	2.36
830	3.97	118		0.50	8.1	0.448	7.33	109	1.42
835	3.96		~1.0 gal	0.42	8.1	0.449	7.28	87	1.78
840	4.0		~1.25 gal	0.38	8.1	0.450	7.22	62	0.98
845	3.97	102		0.30	8.0	0.451	7.23	43	1.26
850	4.02		~1.6 gals	0.26	8.0	0.452	7.20	30	0.99
855	4.12		~1.75 gals	0.34	8.1	0.452	7.16	8	0.92
900	4.08	110	~2.0 gals	0.46	8.0	0.448	7.18	-4	1.03
905	4.09			0.43	8.0	0.447	7.16	-12	0.99
910	4.14		~2.25 gals	0.44	8.0	0.444	7.13	-19	1.34
915	4.17			0.42	8.0	0.441	7.13	-22	0.73
920	4.15		~2.5 gals	0.32	8.0	0.439	7.12	-27	1.20
922	Sample Collected								
926	Restarted	Pump for Post-Sample Collect				Geo Parsons			
931	4.18		~2.75 gals	0.07	8.0	0.438	7.12	-33	0.77

APPENDIX D

POST-REMEDIAL ACTION MONITORING RESULTS (YEARS 1 THROUGH 8)

Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16				
Loc ID								MW16-1		MW16-1		MW16-1		MW16-1		MW16-1		MW16-1		MW16-1				
Matrix								GW		GW		GW		GW		GW		GW		GW				
Sample ID								16LM20000		16LM20001		16LM20013		16LM20014FIL		16LM20014UNFIL		16LM20021FIL		16LM20021UNF				
Sample Date								12/20/2007		12/20/2007		12/9/2008		11/13/2009		11/13/2009		12/16/2010		12/16/2010				
QC Type								SA		DU		SA		SA		SA		SA		SA				
Study ID								LTM		LTM		LTM		LTM		LTM		LTM		LTM				
Sample Round								1		1		2		3		3		4		4				
Filtered								Total		Total		Total		Dissolved		Total		Dissolved		Total				
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual			
Inorganics																								
Aluminum	UG/L	2,400				36	91	61.4	J	91.6	J	148	J	24	U	45	J	23	U	50	U			
Antimony	UG/L	120	GA	3	42	53	91	1	U	1.02		0.95	J	1	U	1	U	2.3	U	2	U			
Arsenic	UG/L	2.7	MCL	10	0	9	91	4.2	U	4.2	U	3.7	U	3.7	U	3.7	U	1.3	U	1.3	U			
Barium	UG/L	600	GA	1,000	0	91	91	60.4		59		125		105		104		110		97	J			
Beryllium	UG/L	0	MCL	4	0	0	91	0.27	U	0.27	U	0.33	U	0.3	U	0.3	U	0.25	U	0.15	U			
Cadmium	UG/L	0.46	GA	5	0	6	91	0.36	U	0.36	U	0.33	U	0.3	U	0.3	U	0.095	U	0.13	U			
Calcium	UG/L	510,000				91	91	107,000	J	105,000	J	176,000		111,000	J	110,000	J	140,000		130,000				
Chromium	UG/L	4.6	GA	50	0	7	91	0.84	U	0.84	U	0.88	U	0.9	U	0.9	U	2.5	U	2.5	U			
Cobalt	UG/L	2				37	91	0.89	U	0.89	U	1.1	U	1.1	U	1.1	U	1.1		1.1				
Copper	UG/L	34.7	GA	200	0	69	91	1.3	U	1.3	U	1.3	U	1.6	J	1.6	J	1.1	U	1.1	U			
Iron	UG/L	4,000	GA	300	24	65	91	35.8	J	68.3		93.3		19	UJ	19	UJ	77	J	100	J			
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	39	J	73		105		1	J	2.4	J	131		152				
Lead	UG/L	88.6	MCL	15	3	42	91	2.9	U	2.9	U	2.9	U	2.9	U	2.9	U	0.2	U	0.5	U			
Magnesium	UG/L	98,000				88	88	16,100	J	15,900	J	25,800		18,000		17,900		21,000		20,000	J			
Manganese	UG/L	631	GA	300	1	85	91	3.3		5		11.8		1	J	2.4	J	54		52				
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.12	U	0.12	U	0.12	U	0.1	U	0.1	U	0.091	U	0.091	U			
Nickel	UG/L	5.5	GA	100	0	54	91	1.2	U	1.2	U	1	U	1.8	J	1.2	J	2.8	J	2.7	J			
Potassium	UG/L	15,000				85	85	886	R	907	R	1,340	J	1,110		1,100		1,200		1,100				
Selenium	UG/L	1.1	GA	10	0	1	91	6.1	U	6.1	U	6.1	U	6.1	U	6.1	U	1	U	1.1	U			
Silver	UG/L	0	GA	50	0	0	91	1	U	1	U	1.3	U	1.3	U	1.3	U	0.25	U	0.18	U			
Sodium	UG/L	550,000	GA	20,000	56	89	89	**24,200**	**J**	**25,300**	**J**	**182,000**		8,000	J	8,000	J	**170,000**	**J**	**160,000**	**J**			
Thallium	UG/L	0.03	MCL	2	0	1	91	0.03	U	0.03	U	0.09	U	0.2	U	0.2	U	0.5	U	0.25	U			
Vanadium	UG/L	2.3				7	91	0.78	U	0.78	U	0.98	U	1	U	1	U	3.8	U	3.2	U			
Zinc	UG/L	34.4				36	91	4.4	J	7.8	J	5.8	J	3.6	U	3.6	U	8.3	U	8.8	J			

Notes:
1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16
Loc ID								MW16-1		MW16-1		MW16-1		MW16-1		MW16-1		MW16-1
Matrix								GW		GW		GW		GW		GW		GW
Sample ID								16LM20028F		16LM20028U		16LM20035F		16LM20035U		16LM20042F		16LM20042U		16LM20049
Sample Date								12/15/2012		12/15/2012		12/17/2013		12/17/2013		12/21/2014		12/21/2014		12/20/2015
QC Type								SA		SA		SA		SA		SA		SA		SA
Study ID								LTM		LTM		LTM		LTM		LTM		LTM		LTM
Sample Round								5		5		6		6		7		7		8
Filtered								Dissolved		Total		Dissolved		Total		Dissolved		Total		Total
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																					
Aluminum	UG/L	2,400				36	91	23	UJ	50	UJ	23	UJ	50	UJ	23	U	50	U	44	J
Antimony	UG/L	120	GA	3	42	53	91	2.3	UJ	2	UJ	2.3	UJ	2	UJ	2.3	U	2	U	0.5	U
Arsenic	UG/L	2.7	MCL	10	0	9	91	1.3	UJ	1.3	UJ	1.3	UJ	1.3	UJ	1.3	U	1.3	U	1.5	U
Barium	UG/L	600	GA	1,000	0	91	91	78	J	78	J	63	J	69	J	99		94		81	
Beryllium	UG/L	0	MCL	4	0	0	91	0.25	UJ	0.15	UJ	0.25	UJ	0.15	UJ	0.25	U	0.15	U	0.17	U
Cadmium	UG/L	0.46	GA	5	0	6	91	0.095	UJ	0.13	UJ	0.095	UJ	0.13	UJ	0.095	U	0.13	U	0.15	U
Calcium	UG/L	510,000				91	91	120,000	J	120,000	J	140,000	J	130,000	J	160,000		150,000		120,000	
Chromium	UG/L	4.6	GA	50	0	7	91	2.5	UJ	2.5	UJ	2.5	UJ	2.5	UJ	2.5	U	2.5	U	3	J
Cobalt	UG/L	2				37	91	0.15	UJ	0.16	J	0.9	J	0.94	J	0.15	U	0.12	U	0.12	J
Copper	UG/L	34.7	GA	200	0	69	91	5.2	J	5	UJ	1.2	J	1.1	UJ	1.3	J	1.9	J	1.7	U
Iron	UG/L	4,000	GA	300	24	65	91	33	UJ	44	UJ	260	J	280	J	33	U	79	J	68	J
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	34	U	46	U	352	J	378	J	11		91		76.7	J
Lead	UG/L	88.6	MCL	15	3	42	91	0.2	UJ	0.5	UJ	0.2	UJ	0.5	UJ	0.2	U	0.5	U	0.98	U
Magnesium	UG/L	98,000				88	88	18,000	J	18,000	J	22,000	J+	22,000	J	25,000		24,000		19,000	
Manganese	UG/L	631	GA	300	1	85	91	1	UJ	2	UJ	92	J	98	J	11		12		8.7	
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.091	UJ	0.091	UJ	0.091	UJ	0.091	UJ	0.091	U	0.091	U	0.08	U
Nickel	UG/L	5.5	GA	100	0	54	91	2.3	J	2	UJ	3.6	J	2	UJ	2	J	2	U	3.8	J
Potassium	UG/L	15,000				85	85	900	J	870	J	810	J	790	J	950		890	J	1,000	
Selenium	UG/L	1.1	GA	10	0	1	91	1	UJ	1.1	UJ	1	UJ	1.1	UJ	1	U	1.1	U	1	U
Silver	UG/L	0	GA	50	0	0	91	0.25	UJ	0.18	UJ	0.25	UJ	0.18	UJ	0.25	U	0.18	U	0.1	U
Sodium	UG/L	550,000	GA	20,000	56	89	89	**63,000**	**J**	**62,000**	**J**	**57,000**	**J**	**60,000**	**J**	**63,000**		**63,000**		**62,000**	
Thallium	UG/L	0.03	MCL	2	0	1	91	0.5	UJ	0.25	UJ	0.5	UJ	0.25	UJ	0.5	U	0.25	U	0.49	U
Vanadium	UG/L	2.3				7	91	3.8	UJ	3.2	UJ	3.8	UJ	3.2	UJ	3.8	U	3.2	U	5.3	U
Zinc	UG/L	34.4				36	91	8.3	UJ	8.4	UJ	8.3	UJ	8.4	UJ	8.3	U	8.4	U	9.6	U

Notes:
1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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3. Shading indicates a concentration above the identified criteria value.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-16 MW16-2 GW 16LM20002 12/20/2007 SA LTM 1 Total		SEAD-16 MW16-2 GW 16LM20007 12/9/2008 SA LTM 2 Total		SEAD-16 MW16-2 GW 16LM20015FIL 11/11/2009 SA LTM 3 Dissolved		SEAD-16 MW16-2 GW 16LM20015UNFIL 11/11/2009 SA LTM 3 Total		SEAD-16 MW16-2 GW 16LM20022FIL 12/15/2010 SA LTM 4 Dissolved		SEAD-16 MW16-2 GW 16LM20022UNF 12/15/2010 SA LTM 4 Total		SEAD-16 MW16-2 GW 16LM20023FIL 12/15/2010 DU LTM 4 Dissolved	
								Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																					
Aluminum	UG/L	2,400				36	91	98.8	J	97.1	J	24	U	205		23	U	50	U	23	U
Antimony	UG/L	120	GA	3	42	53	91	**3.36**		**5.53**		**3.6**		**3.6**		**6.1**		**6.6**		**6.1**	
Arsenic	UG/L	2.7	MCL	10	0	9	91	4.2	U	3.7	U	3.7	U	3.7	U	1.3	U	1.3	U	1.3	U
Barium	UG/L	600	GA	1,000	0	91	91	64.6		69.7		71.9		72.7		68		77	J	67	
Beryllium	UG/L	0	MCL	4	0	0	91	0.27	U	0.33	U	0.3	U	0.3	U	0.25	U	0.15	U	0.25	U
Cadmium	UG/L	0.46	GA	5	0	6	91	0.36	U	0.33	U	0.3	U	0.3	U	0.095	U	0.13	U	0.095	U
Calcium	UG/L	510,000				91	91	143,000	J	138,000		118,000	J	117,000	J	100,000	J	110,000	J	96,000	
Chromium	UG/L	4.6	GA	50	0	7	91	0.84	U	0.88	U	0.9	U	0.9	U	2.5	U	2.5	U	2.5	U
Cobalt	UG/L	2				37	91	0.89	U	1.1	U	1.1	U	1.1	U	0.15	U	0.12	U	0.15	U
Copper	UG/L	34.7	GA	200	0	69	91	4.5	J	4	J	3.4	J	5.1	J	4.4	J	5.9		4.5	J
Iron	UG/L	4,000	GA	300	24	65	91	49.5	J	26.1	J	19	UJ	197	J	33	U	89	J	33	U
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	53	J	27		39.5		260.7	J	12		105		12	
Lead	UG/L	88.6	MCL	15	3	42	91	2.9	U	2.9	U	2.9	U	2.9	U	0.21	J	1.3	J	0.2	U
Magnesium	UG/L	98,000				88	88	15,600	J	15,700		12,600		12,300		12,000		14,000	J	11,000	
Manganese	UG/L	631	GA	300	1	85	91	3.4		0.84	J	39.5		63.7		12		16		12	
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.12	U	0.148	J	0.1	U	0.1	U	0.091	U	0.091	U	0.091	U
Nickel	UG/L	5.5	GA	100	0	54	91	1.2	U	1.6	J	2.2	J	2.6	J	2	U	2	J	2.2	J
Potassium	UG/L	15,000				85	85	2,050	R	2,410	J	3,170		3,140		2,300	J	2,500	J	2,200	J
Selenium	UG/L	1.1	GA	10	0	1	91	6.1	U	6.1	U	6.1	U	6.1	U	1	U	1.1	U	1	U
Silver	UG/L	0	GA	50	0	0	91	1	U	1.3	U	1.3	U	1.3	U	0.25	U	0.18	U	0.25	U
Sodium	UG/L	550,000	GA	20,000	56	89	89	**49,600**	**J**	**63,500**		19,500	J	18,800	J	**33,000**	**J**	**34,000**	**J**	**31,000**	**J**
Thallium	UG/L	0.03	MCL	2	0	1	91	0.03	U	0.09	U	0.2	U	0.2	U	0.5	U	0.25	U	0.5	U
Vanadium	UG/L	2.3				7	91	0.78	U	0.98	U	1	U	1	U	3.8	U	3.2	U	3.8	U
Zinc	UG/L	34.4				36	91	8.2	J	10.2		11.1		11.3		11	J	14	J	12	J

Notes:
1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16				
Loc ID								MW16-2		MW16-2		MW16-2		MW16-2		MW16-2		MW16-2		MW16-2				
Matrix								GW		GW		GW		GW		GW		GW		GW				
Sample ID								16LM20023UNF		16LM20029F		16LM20029U		16LM20036F		16LM20036U		16LM20043F		16LM20043U				
Sample Date								12/15/2010		12/15/2012		12/15/2012		12/16/2013		12/16/2013		12/21/2014		12/21/2014				
QC Type								DU		SA		SA		SA		SA		SA		SA				
Study ID								LTM		LTM		LTM		LTM		LTM		LTM		LTM				
Sample Round								4		5		5		6		6		7		7				
Filtered								Total		Dissolved		Total		Dissolved		Total		Dissolved		Total				
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual			
Inorganics																								
Aluminum	UG/L	2,400				36	91	50	U	23	UJ	50	UJ	23	UJ	50	UJ	23	U	50	U			
Antimony	UG/L	120	GA	3	42	53	91	**6**		**7.8**	**J**	**7.1**	**J**	**3.6**	**J**	**3.2**	**J**	**4.8**	**J**	**4.8**	**J**			
Arsenic	UG/L	2.7	MCL	10	0	9	91	1.3	U	1.3	UJ	1.3	UJ	1.3	UJ	1.3	UJ	1.3	U	1.3	U			
Barium	UG/L	600	GA	1,000	0	91	91	69	J	65	J	62	J	70	J	66	J	72		68				
Beryllium	UG/L	0	MCL	4	0	0	91	0.15	U	0.25	UJ	0.15	UJ	0.25	UJ	0.15	UJ	0.25	U	0.15	U			
Cadmium	UG/L	0.46	GA	5	0	6	91	0.13	U	0.095	UJ	0.13	UJ	0.095	UJ	0.13	UJ	0.095	U	0.13	U			
Calcium	UG/L	510,000				91	91	100,000		110,000	J	100,000	J	120,000	J	100,000	J	110,000		100,000				
Chromium	UG/L	4.6	GA	50	0	7	91	2.5	U	2.5	UJ	2.5	UJ	2.5	UJ	2.5	UJ	2.5	U	2.5	U			
Cobalt	UG/L	2				37	91	0.12	U	0.15	UJ	0.12	UJ	0.23	J	0.23	J	0.15	U	0.12	U			
Copper	UG/L	34.7	GA	200	0	69	91	5.1		4.5	J	5	J	4	J	4.7	J	3.3	J	4.2	J			
Iron	UG/L	4,000	GA	300	24	65	91	63	J	33	UJ	44	UJ	33	UJ	44	UJ	33	J	44	U			
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	76		34	U	46	U	19	J	19	J	34	U	46	U			
Lead	UG/L	88.6	MCL	15	3	42	91	0.97	J	0.24	J	0.66	J	0.38	J	1.1	J	0.2	U	0.87	J			
Magnesium	UG/L	98,000				88	88	12,000	J	13,000	J	11,000	J	14,000	J+	13,000	J	12,000		11,000				
Manganese	UG/L	631	GA	300	1	85	91	13		1	UJ	2	UJ	19	J	19	J	1	U	2	U			
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.091	U	0.091	UJ	0.091	UJ	0.091	UJ	0.091	UJ	0.091	U	0.091	U			
Nickel	UG/L	5.5	GA	100	0	54	91	2.2	J	2.2	J	2	UJ	2	UJ	2	UJ	2.2	J	2.3	J			
Potassium	UG/L	15,000				85	85	2,200	J	2,200	J	1,900	J	1,800	J	1,700	J	1,500		1,400				
Selenium	UG/L	1.1	GA	10	0	1	91	1.1	U	1	UJ	1.1	UJ	1	UJ	1.1	UJ	1	U	1.1	U			
Silver	UG/L	0	GA	50	0	0	91	0.18	U	0.25	UJ	0.18	UJ	0.25	UJ	0.18	UJ	0.25	U	0.18	U			
Sodium	UG/L	550,000	GA	20,000	56	89	89	**32,000**	**J**	20,000	J	17,000	J	**22,000**	**J**	**21,000**	**J**	11,000		9,900				
Thallium	UG/L	0.03	MCL	2	0	1	91	0.25	U	0.5	UJ	0.25	UJ	0.5	UJ	0.25	UJ	0.5	U	0.25	U			
Vanadium	UG/L	2.3				7	91	3.2	U	3.8	UJ	3.2	UJ	3.8	UJ	3.2	UJ	3.8	U	3.2	U			
Zinc	UG/L	34.4				36	91	12	J	9.5	J	8.8	J	24	J	12	J	13	J	12	J			

Notes:
1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-16 MW16-2 GW 16LM20050 12/19/2015 SA LTM 8 Total Value Qual	SEAD-16 MW16-4 GW 16LM20003 12/20/2007 SA LTM 1 Total Value Qual	SEAD-16 MW16-4 GW 16LM20008 12/9/2008 SA LTM 2 Total Value Qual	SEAD-16 MW16-4 GW 16LM20009 12/9/2008 DU LTM 2 Total Value Qual	SEAD-16 MW16-4 GW 16LM20016FIL 11/17/2009 SA LTM 3 Dissolved Value Qual	SEAD-16 MW16-4 GW 16LM20016UNFIL 11/17/2009 SA LTM 3 Total Value Qual	SEAD-16 MW16-4 GW 16LM20024FIL 12/16/2010 SA LTM 4 Dissolved Value Qual
Inorganics														
Aluminum	UG/L	2,400				36	91	58 J	167 J	104 J	101 J	24 U	68 J	23 U
Antimony	UG/L	120	GA	3	42	53	91	2.1 J	**5.11**	2.89	2.94	**6**	**6.3**	2.3 U
Arsenic	UG/L	2.7	MCL	10	0	9	91	1.5 U	4.2 U	3.7 U	3.7 U	3.7 U	3.7 U	1.3 U
Barium	UG/L	600	GA	1,000	0	91	91	94	44.5	290	279	129	123	220
Beryllium	UG/L	0	MCL	4	0	0	91	0.17 U	0.27 U	0.33 U	0.33 U	0.3 U	0.3 U	0.25 U
Cadmium	UG/L	0.46	GA	5	0	6	91	0.15 U	0.36 U	0.33 U	0.33 U	0.3 U	0.3 U	0.095 U
Calcium	UG/L	510,000				91	91	130,000	87,100 J	275,000	267,000	130,000 J	125,000 J	210,000
Chromium	UG/L	4.6	GA	50	0	7	91	1.6 U	1 J	0.88 U	0.88 U	0.9 U	0.9 U	2.5 U
Cobalt	UG/L	2				37	91	0.68	0.89 U	1.1 U	1.1 U	1.8 J	2 J	0.7
Copper	UG/L	34.7	GA	200	0	69	91	3 J	5.4 J	4.4 J	4.2 J	2.4 J	6.2 J	1.4 J
Iron	UG/L	4,000	GA	300	24	65	91	130	95.4	57 J	38.4 J	**329 J**	**419 J**	130 J
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	193	127	65	46 J	417.7 J	**513.5 J**	260
Lead	UG/L	88.6	MCL	15	3	42	91	2.9	2.9 U	2.9 U	2.9 U	2.9 U	2.9 U	0.7 J
Magnesium	UG/L	98,000				88	88	13,000	9,440 R	35,200	34,500	16,800	16,000	31,000
Manganese	UG/L	631	GA	300	1	85	91	63	31.2	7.7	8	88.7	94.5	130
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.08 U	0.12 U	0.12 U	0.12 U	0.1 U	0.1 U	0.091 U
Nickel	UG/L	5.5	GA	100	0	54	91	2.6 J	1.2 U	2.2 J	1.9 J	1.7 J	1.4 J	2.2 J
Potassium	UG/L	15,000				85	85	1,900	1,300 R	3,830 J	3,690 J	3,270	3,270	2,600 J
Selenium	UG/L	1.1	GA	10	0	1	91	1 U	6.1 U	6.1 U	6.1 U	6.1 U	6.1 U	1 U
Silver	UG/L	0	GA	50	0	0	91	0.1 U	1 U	1.3 U	1.3 U	1.3 U	1.3 U	0.25 U
Sodium	UG/L	550,000	GA	20,000	56	89	89	11,000	**40,800 J**	**434,000**	**419,000**	**380,000 J**	**363,000 J**	**540,000 J**
Thallium	UG/L	0.03	MCL	2	0	1	91	0.49 U	0.03 U	0.09 U	0.09 U	0.2 U	0.2 U	0.5 U
Vanadium	UG/L	2.3				7	91	5.3 U	0.78 U	0.98 U	0.98 U	1.1 J	1.1 J	3.8 U
Zinc	UG/L	34.4				36	91	17 J	5.3 J	14.6 J	9.8 J	3.6 U	3.6 U	9.2 J

Notes:
1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-16 MW16-4 GW 16LM20024UNF 12/16/2010 SA LTM 4 Filtered: Total Value	Qual	SEAD-16 MW16-4 GW 16LM20030F 12/15/2012 SA LTM 5 Filtered: Dissolved Value	Qual	SEAD-16 MW16-4 GW 16LM20030U 12/15/2012 SA LTM 5 Filtered: Total Value	Qual	SEAD-16 MW16-4 GW 16LM20037F 12/17/2013 SA LTM 6 Filtered: Dissolved Value	Qual	SEAD-16 MW16-4 GW 16LM20037U 12/17/2013 SA LTM 6 Filtered: Total Value	Qual	SEAD-16 MW16-4 GW 16LM20044F 12/21/2014 SA LTM 7 Filtered: Dissolved Value	Qual	SEAD-16 MW16-4 GW 16LM20044U 12/21/2014 SA LTM 7 Filtered: Total Value	Qual
Inorganics																					
Aluminum	UG/L	2,400				36	91	50	U	23	UJ	50	UJ	23	UJ	50	UJ	23	U	50	U
Antimony	UG/L	120	GA	3	42	53	91	2	U	**4**	**J**	**3.9**	**J**	2.3	UJ	2	UJ	**3.3**	**J**	**3.2**	**J**
Arsenic	UG/L	2.7	MCL	10	0	9	91	1.3	U	1.5	J	1.3	J	1.3	UJ	1.3	UJ	1.3	U	1.3	U
Barium	UG/L	600	GA	1,000	0	91	91	240	J	240	J	230	J	140	J	150	J	170		160	
Beryllium	UG/L	0	MCL	4	0	0	91	0.15	U	0.25	UJ	0.15	UJ	0.25	UJ	0.15	UJ	0.25	U	0.15	U
Cadmium	UG/L	0.46	GA	5	0	6	91	0.13	U	0.095	UJ	0.23	J	0.095	UJ	0.15	J	0.11	J	0.13	U
Calcium	UG/L	510,000				91	91	210,000		230,000	J	220,000	J	210,000	J	190,000	J	220,000		210,000	
Chromium	UG/L	4.6	GA	50	0	7	91	2.5	U	2.5	UJ	2.5	UJ	2.5	UJ	3.6	J	2.5	U	2.5	U
Cobalt	UG/L	2				37	91	0.71		1.9	J	1.9	J	1	J	0.94	J	1.1		1.1	
Copper	UG/L	34.7	GA	200	0	69	91	2.8	J	4.1	J	11	J	1.2	J	1.5	J	4.3	J	5.8	
Iron	UG/L	4,000	GA	300	24	65	91	150	J	130	J	140	J	**350**	**J**	**380**	**J**	170		290	
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	290		270	J	280	J	**580**	**J**	**590**	**J**	370		490	
Lead	UG/L	88.6	MCL	15	3	42	91	3		0.2	UJ	3.4	J	0.28	J	0.65	J	0.27	J	1.5	
Magnesium	UG/L	98,000				88	88	32,000	J	34,000	J	32,000	J	33,000	J+	31,000	J	33,000		32,000	
Manganese	UG/L	631	GA	300	1	85	91	140		140	J	140	J	230	J	210	J	200		200	
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.091	U	0.091	UJ	0.091	UJ	0.091	UJ	0.091	UJ	0.091	U	0.091	U
Nickel	UG/L	5.5	GA	100	0	54	91	2.3	J	2.6	J	3.2	J	3.3	J	2.9	J	4	J	3.5	J
Potassium	UG/L	15,000				85	85	2,600	J	3,200	J	3,100	J	2,500	J	2,400	J	2,000		1,900	
Selenium	UG/L	1.1	GA	10	0	1	91	1.1	U	1	UJ	1.1	UJ	1	UJ	1.1	UJ	1	U	1.1	U
Silver	UG/L	0	GA	50	0	0	91	0.18	U	0.25	UJ	0.18	UJ	0.25	UJ	0.18	UJ	0.25	U	0.18	U
Sodium	UG/L	550,000	GA	20,000	56	89	89	**550,000**	**J**	**340,000**	**J**	**310,000**	**J**	**290,000**	**J**	**270,000**	**J**	**300,000**		**300,000**	
Thallium	UG/L	0.03	MCL	2	0	1	91	0.25	U	0.5	UJ	0.25	UJ	0.5	UJ	0.25	UJ	0.5	U	0.25	U
Vanadium	UG/L	2.3				7	91	3.2	U	3.8	UJ	3.2	UJ	3.8	UJ	3.2	UJ	3.8	U	3.2	U
Zinc	UG/L	34.4				36	91	13	J	12	J	11	J	8.4	UJ	8.4	UJ	14	J	12	J

Notes:
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-16 MW16-4 GW 16LM20051 12/20/2015 SA LTM 8 Total Value	Qual	SEAD-16 MW16-5 GW 16LM20004 12/20/2007 SA LTM 1 Total Value	Qual	SEAD-16 MW16-5 GW 16LM20010 12/10/2008 SA LTM 2 Total Value	Qual	SEAD-16 MW16-5 GW 16LM20017FIL 11/16/2009 SA LTM 3 Dissolved Value	Qual	SEAD-16 MW16-5 GW 16LM20017UNFIL 11/16/2009 SA LTM 3 Total Value	Qual	SEAD-16 MW16-5 GW 16LM20025FIL 12/15/2010 SA LTM 4 Dissolved Value	Qual	SEAD-16 MW16-5 GW 16LM20025UNF 12/15/2010 SA LTM 4 Total Value	Qual
Inorganics																					
Aluminum	UG/L	2,400				36	91	18	U	160	J	563		24	U	164	J	23	U	160	
Antimony	UG/L	120	GA	3	42	53	91	2	J	1.82		**4.23**		1	U	1	U	2.3	U	2	U
Arsenic	UG/L	2.7	MCL	10	0	9	91	1.5	U	4.2	U	3.7	U	3.7	U	3.7	U	1.3	U	1.3	U
Barium	UG/L	600	GA	1,000	0	91	91	140		38.9		22		42.8		42		34		33	J
Beryllium	UG/L	0	MCL	4	0	0	91	0.17	U	0.27	U	0.33	U	0.3	U	0.3	U	0.25	U	0.15	U
Cadmium	UG/L	0.46	GA	5	0	6	91	0.34	J	0.36	U	0.33	U	0.3	U	0.3	U	0.095	U	0.13	U
Calcium	UG/L	510,000				91	91	160,000		89,000	J	53,100		115,000	J	110,000	J	90,000		86,000	
Chromium	UG/L	4.6	GA	50	0	7	91	1.6	U	1.1	J	1.2	J	0.9	U	0.9	U	2.5	U	2.5	U
Cobalt	UG/L	2				37	91	0.28	J	0.89	U	1.1	U	1.1	U	1.1	U	0.15	U	0.12	U
Copper	UG/L	34.7	GA	200	0	69	91	6.8		3.1	J	10.6		1.3	U	1.3	U	1.1	U	1.1	U
Iron	UG/L	4,000	GA	300	24	65	91	33	J	**1,200**		**699**		**800**	**J**	**1,150**	**J**	**480**	**J**	**660**	**J**
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	85	J	**1,238**		**731**		**970**	**J**	**1,323**	**J**	**680**		**820**	
Lead	UG/L	88.6	MCL	15	3	42	91	1.1	J	2.9	U	10.1		2.9	U	2.9	U	0.2	U	0.77	J
Magnesium	UG/L	98,000				88	88	25,000		9,380	R	6,050		12,200		11,800		10,000		9,700	J
Manganese	UG/L	631	GA	300	1	85	91	52		37.6		32.4		170		173		200		160	
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.08	U	0.12	U	0.12	U	0.1	U	0.1	U	0.091	U	0.091	U
Nickel	UG/L	5.5	GA	100	0	54	91	3.7	J	1.2	U	2.6	J	1.8	J	2	J	2	U	2	U
Potassium	UG/L	15,000				85	85	1,900		4,420	R	2,610	J	2,370		2,380		2,200	J	2,100	J
Selenium	UG/L	1.1	GA	10	0	1	91	1	U	6.1	U	6.1	U	6.1	U	6.1	U	1	U	1.1	U
Silver	UG/L	0	GA	50	0	0	91	0.1	U	1	U	1.3	U	1.3	U	1.3	U	0.25	U	0.18	U
Sodium	UG/L	550,000	GA	20,000	56	89	89	**250,000**		8,410	R	2,180		2,700	J	2,800	J	1,800	J	1,800	J
Thallium	UG/L	0.03	MCL	2	0	1	91	0.49	U	0.03	U	0.09	U	0.2	U	0.2	U	0.5	U	0.25	U
Vanadium	UG/L	2.3				7	91	5.3	U	1.2	J	2.3	J	1	U	1.1	J	3.8	U	3.2	U
Zinc	UG/L	34.4				36	91	16	J	34.4		10.3		3.6	U	3.6	U	8.3	U	8.4	U

Notes:
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Appendix D
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Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16	
Loc ID								MW16-5		MW16-5		MW16-5		MW16-5		MW16-5		MW16-5	
Matrix								GW		GW		GW		GW		GW		GW	
Sample ID								16LM20031F		16LM20031U		16LM20038F		16LM20038U		16LM20039F		16LM20045F	
Sample Date								12/15/2012		12/15/2012		12/16/2013		12/16/2013		12/17/2013		12/20/2014	
QC Type								SA		SA		SA		SA		DU		SA	
Study ID								LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round								5		5		6		6		6		7	
Filtered								Dissolved		Total		Dissolved		Total		Dissolved		Total		Dissolved	
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																					
Aluminum	UG/L	2,400				36	91	23	UJ	50	UJ	23	UJ	50	UJ	23	UJ	50	UJ	23	U
Antimony	UG/L	120	GA	3	42	53	91	2.3	UJ	2	UJ	2.3	UJ	2	UJ	2.3	UJ	2	UJ	2.3	U
Arsenic	UG/L	2.7	MCL	10	0	9	91	2.6	J	2.7	J	1.3	UJ	1.3	UJ	1.3	UJ	1.3	UJ	1.8	J
Barium	UG/L	600	GA	1,000	0	91	91	34	J	39	J	40	J	38	J	41	J	41	J	49	J
Beryllium	UG/L	0	MCL	4	0	0	91	0.25	UJ	0.15	UJ	0.25	UJ	0.15	UJ	0.25	UJ	0.15	UJ	0.25	U
Cadmium	UG/L	0.46	GA	5	0	6	91	0.095	UJ	0.13	UJ	0.095	UJ	0.13	UJ	0.095	UJ	0.13	UJ	0.095	U
Calcium	UG/L	510,000				91	91	97,000	J	96,000	J	100,000	J	88,000	J	110,000	J	95,000	J	110,000	
Chromium	UG/L	4.6	GA	50	0	7	91	2.5	UJ	2.5	UJ	2.5	UJ	2.5	UJ	2.5	UJ	2.5	UJ	2.5	U
Cobalt	UG/L	2				37	91	0.22	J	0.23	J	0.15	UJ	0.12	UJ	0.15	UJ	0.12	UJ	0.15	U
Copper	UG/L	34.7	GA	200	0	69	91	1.1	J	5	UJ	1.1	UJ	1.1	UJ	1.1	UJ	1.1	UJ	1.1	U
Iron	UG/L	4,000	GA	300	24	65	91	**1,100**	**J**	**1,300**	**J**	**440**	**J**	**510**	**J**	**490**	**J**	**530**	**J**	**360**	**J**
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	**1,230**	**J**	**1,430**	**J**	**670**	**J**	**680**	**J**	**710**	**J**	**720**	**J**	**520**	**J**
Lead	UG/L	88.6	MCL	15	3	42	91	0.2	UJ	0.5	UJ	0.2	UJ	0.5	UJ	0.22	J	0.5	UJ	0.2	U
Magnesium	UG/L	98,000				88	88	9,900	J	9,800	J	10,000	J+	9,500	J	11,000	J+	10,000	J	11,000	
Manganese	UG/L	631	GA	300	1	85	91	130	J	130	J	230	J	170	J	220	J	190	J	160	J
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.1	J	0.091	UJ	0.091	UJ	0.091	UJ	0.091	UJ	0.091	UJ	0.091	U
Nickel	UG/L	5.5	GA	100	0	54	91	2.1	J	2	UJ	2	UJ	2	UJ	2	UJ	2	UJ	2.1	J
Potassium	UG/L	15,000				85	85	2,100	J	2,100	J	2,300	J	1,900	J	2,300	J	2,100	J	3,500	J
Selenium	UG/L	1.1	GA	10	0	1	91	1	UJ	1.1	UJ	1	UJ	1.1	UJ	1	UJ	1.1	UJ	1	U
Silver	UG/L	0	GA	50	0	0	91	0.25	UJ	0.18	UJ	0.25	UJ	0.18	UJ	0.25	UJ	0.18	UJ	0.25	U
Sodium	UG/L	550,000	GA	20,000	56	89	89	1,600	J	1,500	J	1,400	J	1,300	J	1,400	J	1,300	J	1,900	
Thallium	UG/L	0.03	MCL	2	0	1	91	0.5	UJ	0.25	UJ	0.5	UJ	0.25	UJ	0.5	UJ	0.25	UJ	0.5	U
Vanadium	UG/L	2.3				7	91	3.8	UJ	3.2	UJ	3.8	UJ	3.2	UJ	3.8	UJ	3.2	UJ	3.8	U
Zinc	UG/L	34.4				36	91	8.3	UJ	8.4	UJ	8.3	UJ	8.4	UJ	8.3	UJ	8.4	UJ	8.3	U

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Seneca Army Depot Activity

Area								SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16				
Loc ID								MW16-5		MW16-5		MW16-6		MW16-6		MW16-6		MW16-6		MW16-6				
Matrix								GW		GW		GW		GW		GW		GW		GW				
Sample ID								16LM20045U		16LM20052		16LM20005		16LM20011		16LM20018FIL		16LM20018UNFIL		16LM20026FIL				
Sample Date								12/20/2014		12/19/2015		12/20/2007		12/9/2008		11/17/2009		11/17/2009		12/15/2010				
QC Type								SA		SA		SA		SA		SA		SA		SA				
Study ID								LTM		LTM		LTM		LTM		LTM		LTM		LTM				
Sample Round								7		8		1		2		3		3		4				
Filtered								Total		Total		Total		Total		Dissolved		Total		Dissolved				
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual			
Inorganics																								
Aluminum	UG/L	2,400				36	91	53	J	31	J	168	J	189	J	107	J	442		23	U			
Antimony	UG/L	120	GA	3	42	53	91	2	U	0.75	J	1	U	0.92	J	0.9	J	1	U	2.3	U			
Arsenic	UG/L	2.7	MCL	10	0	9	91	1.3	J	1.5	U	4.2	U	3.7	U	3.7	U	3.7	U	1.3	U			
Barium	UG/L	600	GA	1,000	0	91	91	40	J	41		31.8		39.1		78.5		80.2		44				
Beryllium	UG/L	0	MCL	4	0	0	91	0.15	U	0.17	U	0.27	U	0.33	U	0.3	U	0.3	U	0.25	U			
Cadmium	UG/L	0.46	GA	5	0	6	91	0.13	U	0.15	U	0.36	U	0.33	U	0.3	U	0.3	U	0.095	U			
Calcium	UG/L	510,000				91	91	92,000		110,000		80,400	J	84,300		112,000	J	112,000	J	68,000				
Chromium	UG/L	4.6	GA	50	0	7	91	2.5	U	1.6	U	0.84	U	0.88	U	0.9	U	0.9	U	2.5	U			
Cobalt	UG/L	2				37	91	0.12	U	0.12	U	0.89	U	1.1	U	1.1	U	1.1	U	0.15	U			
Copper	UG/L	34.7	GA	200	0	69	91	3.1	J	1.7	U	3.4	J	2.1	J	1.9	J	2.5	J	1.5	J			
Iron	UG/L	4,000	GA	300	24	65	91	280	J	**570**		**418**		153		55	J	**440**	**J**	33	U			
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	410	J	710		441		158		153.4	J	**515**	**J**	2.1	J			
Lead	UG/L	88.6	MCL	15	3	42	91	0.5	U	0.98	U	2.9	U	2.9	U	2.9	U	2.9	U	0.2	U			
Magnesium	UG/L	98,000				88	88	9,000		10,000		7,100	R	7,380		9,970		9,950		6,600				
Manganese	UG/L	631	GA	300	1	85	91	130	J	140		23.3		4.8		98.4		75		2.1	J			
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.091	U	0.08	U	0.12	U	0.12	U	0.1	U	0.1	U	0.091	U			
Nickel	UG/L	5.5	GA	100	0	54	91	2.3	J	2.3	J	1.2	U	1	U	1.2	J	2.6	J	2	U			
Potassium	UG/L	15,000				85	85	2,800	J	2,500		2,690	R	2,310	J	2,380		2,580		1,500				
Selenium	UG/L	1.1	GA	10	0	1	91	1.1	U	1	U	6.1	U	6.1	U	6.1	U	6.1	U	1	U			
Silver	UG/L	0	GA	50	0	0	91	0.18	U	0.1	U	1	U	1.3	U	1.3	U	1.3	U	0.25	U			
Sodium	UG/L	550,000	GA	20,000	56	89	89	1,600		1,800		6,110	R	9,200		**22,000**	**J**	**20,600**	**J**	7,600	J			
Thallium	UG/L	0.03	MCL	2	0	1	91	0.25	U	0.49	U	0.03	U	0.09	U	0.008	U	0.008	U	0.5	U			
Vanadium	UG/L	2.3				7	91	3.2	U	5.3	U	0.86	J	0.98	U	1	U	1.3	J	3.8	U			
Zinc	UG/L	34.4				36	91	8.4	U	9.6	U	5.5	J	3.7	J	3.6	U	3.6	U	8.3	U			

Notes:
1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-16 MW16-6 GW 16LM20026UNF 12/15/2010 SA LTM 4 Filtered: Total Value	Qual	SEAD-16 MW16-6 GW 16LM20032F 12/15/2012 SA LTM 5 Filtered: Dissolved Value	Qual	SEAD-16 MW16-6 GW 16LM20032U 12/15/2012 SA LTM 5 Filtered: Total Value	Qual	SEAD-16 MW16-6 GW 16LM20040F 12/17/2013 SA LTM 6 Filtered: Dissolved Value	Qual	SEAD-16 MW16-6 GW 16LM20040U 12/17/2013 SA LTM 6 Filtered: Total Value	Qual	SEAD-16 MW16-6 GW 16LM20046F 12/21/2014 SA LTM 7 Filtered: Dissolved Value	Qual	SEAD-16 MW16-6 GW 16LM20046U 12/21/2014 SA LTM 7 Filtered: Total Value	Qual
Inorganics																					
Aluminum	UG/L	2,400				36	91	61	J	23	UJ	300	J	23	UJ	50	UJ	23	U	140	
Antimony	UG/L	120	GA	3	42	53	91	2	U	2.3	UJ	2	UJ	2.3	UJ	2	UJ	2.3	U	2	U
Arsenic	UG/L	2.7	MCL	10	0	9	91	1.3	U	1.3	UJ	1.3	J	1.3	UJ	1.3	UJ	1.3	U	1.3	U
Barium	UG/L	600	GA	1,000	0	91	91	50	J	41	J	45	J	53	J	58	J	58		58	
Beryllium	UG/L	0	MCL	4	0	0	91	0.15	U	0.25	UJ	0.15	UJ	0.25	UJ	0.15	UJ	0.25	U	0.15	U
Cadmium	UG/L	0.46	GA	5	0	6	91	0.13	U	0.095	UJ	0.13	UJ	0.095	UJ	0.13	UJ	0.095	U	0.13	U
Calcium	UG/L	510,000				91	91	78,000		70,000	J	74,000	J	92,000	J	84,000	J	83,000		83,000	
Chromium	UG/L	4.6	GA	50	0	7	91	2.5	U	2.5	UJ	2.5	UJ	2.5	UJ	2.5	UJ	2.5	U	2.5	U
Cobalt	UG/L	2				37	91	0.12	U	0.18	J	0.43	J	0.35	J	0.34	J	0.15	U	0.12	U
Copper	UG/L	34.7	GA	200	0	69	91	2	J	4.5	J	5	UJ	1.1	UJ	1.1	UJ	2.3	J	2.8	J
Iron	UG/L	4,000	GA	300	24	65	91	110	J	33	J	**790**	**J**	180	J	210	J	57	J	140	
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	113.5	J	43	J	**816**	**J**	340	J	360	J	58.8	J	148.4	
Lead	UG/L	88.6	MCL	15	3	42	91	0.5	U	0.2	UJ	0.5	UJ	0.2	UJ	0.54	J	0.2	U	0.5	U
Magnesium	UG/L	98,000				88	88	7,600	J	7,200	J	7,600	J	9,500	J+	9,500	J	8,300		8,500	
Manganese	UG/L	631	GA	300	1	85	91	3.5	J	10	J	26	J	160	J	150	J	1.8	J	8.4	
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.091	U	0.091	UJ	0.091	UJ	0.091	UJ	0.091	UJ	0.091	U	0.091	U
Nickel	UG/L	5.5	GA	100	0	54	91	2	U	2	UJ	2	J	2	UJ	2	UJ	2.2	J	2	U
Potassium	UG/L	15,000				85	85	1,800		2,400	J	2,400	J	1,900	J	1,800	J	2,100		2,000	
Selenium	UG/L	1.1	GA	10	0	1	91	1.1	U	1	UJ	1.1	UJ	1	UJ	1.1	UJ	1	U	1.1	U
Silver	UG/L	0	GA	50	0	0	91	0.18	U	0.25	UJ	0.18	UJ	0.25	UJ	0.18	UJ	0.25	U	0.18	U
Sodium	UG/L	550,000	GA	20,000	56	89	89	8,400	J	8,700	J	8,000	J	14,000	J	13,000	J	8,500		8,300	
Thallium	UG/L	0.03	MCL	2	0	1	91	0.25	U	0.5	UJ	0.25	UJ	0.5	UJ	0.25	UJ	0.5	U	0.25	U
Vanadium	UG/L	2.3				7	91	3.2	U	3.8	UJ	3.2	UJ	3.8	UJ	3.2	UJ	3.8	U	3.2	U
Zinc	UG/L	34.4				36	91	8.4	U	8.3	UJ	8.4	UJ	8.3	UJ	8.4	UJ	8.3	U	8.4	U

Notes:
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	SEAD-16 MW16-6 GW 16LM20053 12/19/2015 SA LTM 8 Total Value Qual	SEAD-16 MW16-7 GW 16LM20006 12/20/2007 SA LTM 1 Total Value Qual	SEAD-16 MW16-7 GW 16LM20012 12/10/2008 SA LTM 2 Total Value Qual	SEAD-16 MW16-7 GW 16LM20019FIL 11/12/2009 SA LTM 3 Dissolved Value Qual	SEAD-16 MW16-7 GW 16LM20019UNFIL 11/12/2009 SA LTM 3 Total Value Qual	SEAD-16 MW16-7 GW 16LM20020FIL 11/12/2009 DU LTM 3 Dissolved Value Qual	SEAD-16 MW16-7 GW 16LM20020UNFIL 11/12/2009 DU LTM 3 Total Value Qual
Inorganics														
Aluminum	UG/L	2,400				36	91	2,400	45.9 J	577	32 J	182 J	25 J	116 J
Antimony	UG/L	120	GA	3	42	53	91	1 J	**9.58**	**13.6**	**15.2**	**15.7**	**13.9**	**16.3**
Arsenic	UG/L	2.7	MCL	10	0	9	91	1.9 J	4.2 U	3.7 U	3.7 U	3.7 U	3.7 U	3.7 U
Barium	UG/L	600	GA	1,000	0	91	91	73	170	122	83.6	81.6	83.9	80.3
Beryllium	UG/L	0	MCL	4	0	0	91	0.17 U	0.27 U	0.33 U	0.3 U	0.3 U	0.3 U	0.3 U
Cadmium	UG/L	0.46	GA	5	0	6	91	0.33 J	0.46 J	0.33 U	0.3 U	0.3 U	0.3 U	0.3 U
Calcium	UG/L	510,000				91	91	80,000	194,000	133,000	85,000 J	84,600 J	81,900 J	82,800 J
Chromium	UG/L	4.6	GA	50	0	7	91	4.6 J	0.84 U	1.6 J	0.9 U	0.9 U	0.9 U	0.9 U
Cobalt	UG/L	2				37	91	1.6	1.6 J	1.1 J	1.1 U	1.1 U	1.1 U	1.1 U
Copper	UG/L	34.7	GA	200	0	69	91	6.3	34.7	20.2	3.1 J	5 J	3.5 J	4.1 J
Iron	UG/L	4,000	GA	300	24	65	91	**4,000**	29.2 J	**770**	19 UJ	135 J	19 UJ	61 J
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	**4,120**	**660 J**	**990**	136	244 J	152	168 J
Lead	UG/L	88.6	MCL	15	3	42	91	2.2 J	**26.5**	**88.6**	4.4 J	12.1	4.9 J	9.4
Magnesium	UG/L	98,000				88	88	8,300	32,000 J	25,100	15,900	16,500	14,800	16,200
Manganese	UG/L	631	GA	300	1	85	91	120	**631**	220	136	109	152	107
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.08 U	0.507	0.12 U	0.1 U	0.1 U	0.1 U	0.1 U
Nickel	UG/L	5.5	GA	100	0	54	91	5.1	5.5 J	2.6 J	1.9 J	1.7 J	2 J	1.1 J
Potassium	UG/L	15,000				85	85	2,600	5,480 J	5,670 J	6,520	5,780	7,010	5,630
Selenium	UG/L	1.1	GA	10	0	1	91	1.1 J	6.1 U	6.1 U	6.1 U	6.1 U	6.1 U	6.1 U
Silver	UG/L	0	GA	50	0	0	91	0.1 U	1 U	1.3 U	1.3 U	1.3 U	1.3 U	1.3 U
Sodium	UG/L	550,000	GA	20,000	56	89	89	10,000	**68,400 J**	**74,900**	**52,100 J**	**47,100 J**	**55,900 J**	**46,100 J**
Thallium	UG/L	0.03	MCL	2	0	1	91	0.49 U	0.03 J	0.09 U	0.2 U	0.2 U	0.2 U	0.2 U
Vanadium	UG/L	2.3				7	91	5.3 U	0.78 U	0.98 U	1 U	1 U	1 U	1 U
Zinc	UG/L	34.4				36	91	18 J	3.6 U	8.6 J	3.6 U	3.6 U	3.6 U	3.6 U

Notes:
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
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Area								SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16	
Loc ID								MW16-7		MW16-7		MW16-7		MW16-7		MW16-7		MW16-7	
Matrix								GW		GW		GW		GW		GW		GW	
Sample ID								16LM20027FIL		16LM20027UNF		16LM20033F		16LM20033U		16LM20034F		16LM20041F	
Sample Date								12/15/2010		12/15/2010		12/15/2012		12/15/2012		12/15/2012		12/17/2013	
QC Type								SA		SA		SA		SA		DU		SA	
Study ID								LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round								4		4		5		5		5		6	
Filtered								Dissolved		Total		Dissolved		Total		Dissolved		Dissolved	
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																			
Aluminum	UG/L	2,400				36	91	23	U	50	U	23	UJ	50	UJ	23	UJ	50	UJ	23	UJ
Antimony	UG/L	120	GA	3	42	53	91	**15**		**16**		**13**	**J**	**13**	**J**	**13**	**J**	**14**	**J**	**16**	**J**
Arsenic	UG/L	2.7	MCL	10	0	9	91	1.3	U	1.3	U	1.3	J	1.3	UJ	1.3	UJ	1.3	UJ	1.3	UJ
Barium	UG/L	600	GA	1,000	0	91	91	69		71	J	100	J	100	J	99	J	100	J	100	J
Beryllium	UG/L	0	MCL	4	0	0	91	0.25	U	0.15	U	0.25	UJ	0.15	UJ	0.25	UJ	0.15	UJ	0.25	UJ
Cadmium	UG/L	0.46	GA	5	0	6	91	0.095	U	0.13	U	0.095	UJ	0.13	UJ	0.095	UJ	0.13	UJ	0.095	UJ
Calcium	UG/L	510,000				91	91	82,000		86,000		110,000	J	100,000	J	100,000	J	110,000	J	120,000	J
Chromium	UG/L	4.6	GA	50	0	7	91	2.5	U	2.5	U	2.5	UJ	2.5	UJ	2.5	UJ	2.5	UJ	2.5	UJ
Cobalt	UG/L	2				37	91	0.15	U	0.12	U	0.23	J	0.22	J	0.24	J	0.24	J	0.19	J
Copper	UG/L	34.7	GA	200	0	69	91	1.8	J	2.7	J	4.1	J	8.3	J	1.7	J	5.6	J	3.4	J
Iron	UG/L	4,000	GA	300	24	65	91	33	U	45	J	33	UJ	44	UJ	33	UJ	44	UJ	33	UJ
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	35		79		92	J	90	J	98	J	91	J	16	J
Lead	UG/L	88.6	MCL	15	3	42	91	1	J	6.3		1.3	J	2.5	J	2.3	J	2.6	J	1.9	J
Magnesium	UG/L	98,000				88	88	18,000		19,000	J	21,000	J	21,000	J	20,000	J	22,000	J	26,000	J+
Manganese	UG/L	631	GA	300	1	85	91	35		34		92	J	90	J	98	J	91	J	16	J
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.091	U	0.091	U	0.091	UJ	0.091	UJ	0.091	UJ	0.091	UJ	0.091	UJ
Nickel	UG/L	5.5	GA	100	0	54	91	2	U	2	U	2	UJ	2.2	J	2	UJ	2.4	J	2	UJ
Potassium	UG/L	15,000				85	85	2,800	J	2,700	J	5,300	J	5,200	J	5,100	J	5,400	J	3,100	J
Selenium	UG/L	1.1	GA	10	0	1	91	1	U	1.1	U	1	UJ	1.1	UJ	1	UJ	1.1	UJ	1	UJ
Silver	UG/L	0	GA	50	0	0	91	0.25	U	0.18	U	0.25	UJ	0.18	UJ	0.25	UJ	0.18	UJ	0.25	UJ
Sodium	UG/L	550,000	GA	20,000	56	89	89	**29,000**	**J**	**28,000**	**J**	**35,000**	**J**	**32,000**	**J**	**33,000**	**J**	**32,000**	**J**	**28,000**	**J**
Thallium	UG/L	0.03	MCL	2	0	1	91	0.5	U	0.25	U	0.5	UJ	0.25	UJ	0.5	UJ	0.25	UJ	0.5	UJ
Vanadium	UG/L	2.3				7	91	3.8	U	3.2	U	3.8	UJ	3.2	UJ	3.8	UJ	3.2	UJ	3.8	UJ
Zinc	UG/L	34.4				36	91	8.3	U	8.4	U	8.3	UJ	8.4	UJ	8.3	UJ	8.4	UJ	8.3	UJ

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Annual Report - SEAD 16 and SEAD 17
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Area								SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16		SEAD-16	
Loc ID								MW16-7		MW16-7		MW16-7		MW16-7		MW16-7		MW16-7	
Matrix								GW		GW		GW		GW		GW		GW	
Sample ID								16LM20041U		16LM20047F		16LM20047U		16LM20048F		16LM20048U		16LM20054	16LM20055
Sample Date								12/17/2013		12/20/2014		12/20/2014		12/20/2014		12/20/2014		12/19/2015	12/19/2015
QC Type								SA		SA		SA		DU		DU		SA	DU
Study ID								LTM		LTM		LTM		LTM		LTM		LTM	LTM
Sample Round								6		7		7		7		7		8	8
Filtered								Total		Dissolved		Total		Dissolved		Total		Total	Total
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																					
Aluminum	UG/L	2,400				36	91	50	UJ	29	J	50	U	23	U	50	U	140	J	36	J
Antimony	UG/L	120	GA	3	42	53	91	**15**	**J**	**16**		**15**		**15**		**14**		**120**	**J**	**19**	**J**
Arsenic	UG/L	2.7	MCL	10	0	9	91	1.3	UJ	1.3	U	1.3	U	1.3	U	1.3	U	7.5	U	1.5	U
Barium	UG/L	600	GA	1,000	0	91	91	100	J	110		95		110		100		600	J	130	J
Beryllium	UG/L	0	MCL	4	0	0	91	0.15	UJ	0.25	U	0.15	U	0.25	U	0.15	U	0.85	U	0.17	U
Cadmium	UG/L	0.46	GA	5	0	6	91	0.13	UJ	0.095	U	0.13	U	0.095	U	0.13	U	0.15	U	0.15	U
Calcium	UG/L	510,000				91	91	110,000	J	110,000		100,000		110,000		110,000		510,000	J	110,000	J
Chromium	UG/L	4.6	GA	50	0	7	91	2.5	UJ	2.5	U	2.5	U	2.5	U	2.5	U	8	U	1.6	U
Cobalt	UG/L	2				37	91	0.2	J	0.25	J	0.12	U	0.15	U	0.12	U	0.6	U	0.12	J
Copper	UG/L	34.7	GA	200	0	69	91	2.5	J	3.2	J	3.6	J	3.3	J	3.8	J	21	J	4.2	J
Iron	UG/L	4,000	GA	300	24	65	91	44	UJ	52	J	44	U	33	U	44	U	**370**	**J**	62	J
Iron+Manganese	UG/L	1,430	GA	500	20	78	84	15	J	80	J	23	J	38	J	33	J	396	J	69.4	J
Lead	UG/L	88.6	MCL	15	3	42	91	6	J	1.8		4.2		1.8		4.1		**48**	**J**	10	J
Magnesium	UG/L	98,000				88	88	27,000	J	23,000		22,000		23,000		21,000		98,000	J	20,000	J
Manganese	UG/L	631	GA	300	1	85	91	15	J	28	J	23	J	38	J	33	J	26	J	7.4	J
Mercury	UG/L	0.507	GA	0.7	0	3	91	0.091	UJ	0.091	U	0.091	U	0.091	U	0.091	U	0.08	U	0.08	U
Nickel	UG/L	5.5	GA	100	0	54	91	2	UJ	3.2	J	2.4	J	2	J	2	U	9.5	U	1.9	U
Potassium	UG/L	15,000				85	85	2,900	J	3,700		3,500		4,600		3,900		15,000	J	3,600	J
Selenium	UG/L	1.1	GA	10	0	1	91	1.1	UJ	1	U	1.1	U	1	U	1.1	U	5	U	1	U
Silver	UG/L	0	GA	50	0	0	91	0.18	UJ	0.25	U	0.18	U	0.25	U	0.18	U	0.5	U	0.1	U
Sodium	UG/L	550,000	GA	20,000	56	89	89	**27,000**	**J**	**30,000**		**29,000**		**36,000**		**33,000**		**89,000**	**J**	**23,000**	**J**
Thallium	UG/L	0.03	MCL	2	0	1	91	0.25	UJ	0.5	U	0.25	U	0.5	U	0.25	U	2.5	U	0.49	U
Vanadium	UG/L	2.3				7	91	3.2	UJ	3.8	U	3.2	U	3.8	U	3.2	U	27	U	5.3	U
Zinc	UG/L	34.4				36	91	8.4	UJ	8.3	U	8.4	U	8.7	J	8.4	U	48	U	9.6	U

Notes:
1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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   J = the reported value is an estimated concentration
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3. Shading indicates a concentration above the identified criteria value.
   SA = Sample
   DU = Duplicate Sample
4. Rejected values are not included in the number of samples analyzed.

Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17				
Loc ID								MW17-1		MW17-1		MW17-1		MW17-1		MW17-1		MW17-1		MW17-1				
Matrix								GW		GW		GW		GW		GW		GW		GW				
Sample ID								17LM20000		17LM20005		17LM20010FIL		17LM20010UNFIL		17LM20016FIL		17LM20016UNF		17LM20020F				
Sample Date								12/20/2007		12/11/2008		11/18/2009		11/18/2009		12/17/2010		12/17/2010		12/11/2012				
QC Type								SA		SA		SA		SA		SA		SA		SA				
Study ID								LTM		LTM		LTM		LTM		LTM		LTM		LTM				
Sample Round								1		2		3		3		4		4		5				
Filtered								Total		Total		Dissolved		Total		Dissolved		Total		Dissolved				
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual			
Inorganics																								
Aluminum	UG/L	19,600				25	65	204		219		37	J	59	J	23	U	50	U	23	UJ			
Antimony	UG/L	4.4	GA	3	6	16	65	1	U	1	U	1	U	1	U	2.3	U	2	U	2.3	UJ			
Arsenic	UG/L	7.8	MCL	10	0	2	65	4.2	U	3.7	U	3.7	U	3.7	U	1.3	U	1.3	U	1.3	UJ			
Barium	UG/L	251	GA	1,000	0	65	65	70		79		99.1		99		61		63	J	28	J			
Beryllium	UG/L	1.2	MCL	4	0	1	65	0.27	U	0.33	U	0.3	U	0.3	U	0.25	U	0.15	U	0.25	UJ			
Cadmium	UG/L	1.7	GA	5	0	4	65	0.36	U	0.33	U	0.3	U	0.3	U	0.095	U	0.13	U	0.095	UJ			
Calcium	UG/L	195,000				65	65	98,300	J	95,600		109,000	J	108,000	J	96,000		100,000		53,000	J			
Chromium	UG/L	37.2	GA	50	0	4	65	0.84	U	0.88	U	0.9	U	0.9	U	2.5	U	2.5	U	2.5	UJ			
Cobalt	UG/L	10.5				43	65	0.89	U	1.1	U	1.1	U	1.1	U	0.15	U	0.3	J	0.32	J			
Copper	UG/L	46.7	GA	200	0	33	65	1.3	U	1.3	U	1.3	U	1.3	U	1.1	U	1.1	J	4.7	J			
Iron	UG/L	25,500	GA	300	15	51	65	106		126		19	UJ	42	J	33	U	270	J	47	J			
Iron+Manganese	UG/L	25,929	GA	500	13	56	60	119		141		38.9		67.6	J	4.2	J	312		54.2	J			
Lead	UG/L	103	MCL	15	1	11	65	2.9	U	2.9	U	2.9	U	2.9	U	0.2	U	0.5	U	0.2	UJ			
Magnesium	UG/L	27,300				62	62	21,800	J	20,600		24,300		24,000		19,000		20,000	J	7,200	J			
Manganese	UG/L	911	GA	300	2	60	65	13.2		14.9		38.9		25.6		4.2	J	42		7.2	J			
Mercury	UG/L	0.14	GA	0.7	0	2	65	0.12	U	0.12	U	0.1	U	0.1	U	0.091	U	0.091	U	0.14	J			
Nickel	UG/L	34	GA	100	0	22	65	1.2	U	1.3	J	1	U	1	U	2	U	2	U	2	UJ			
Potassium	UG/L	7,810				59	60	614	R	462	J	260	J	254	J	690		690	J	380	J			
Selenium	UG/L	0	GA	10	0	0	65	6.1	U	6.1	U	6.1	U	6.1	U	1	U	1.1	U	1	UJ			
Silver	UG/L	0	GA	50	0	0	65	1	U	1.3	U	1.3	U	1.3	U	0.25	U	0.18	U	0.25	UJ			
Sodium	UG/L	366,000	GA	20,000	4	61	61	7,790	R	8,380		7,300	J	7,400	J	6,000	J	6,200	J	2,400	J			
Thallium	UG/L	0.08	MCL	2	0	2	65	0.03	U	0.09	U	0.008	U	0.008	U	0.5	U	0.25	U	0.5	UJ			
Vanadium	UG/L	32.8				2	65	0.78	U	0.98	U	1	U	1	U	3.8	U	3.2	U	3.8	UJ			
Zinc	UG/L	935				35	65	4.7	J	4	J	3.6	U	3.6	U	8.3	U	8.4	U	8.3	UJ			

Notes:

1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17	
Loc ID								MW17-1		MW17-1		MW17-1		MW17-1		MW17-1		MW17-1		MW17-2			
Matrix								GW		GW		GW		GW		GW		GW		GW			
Sample ID								17LM20020U		17LM20025F		17LM20025U		17LM20030F		17LM20030U		17LM20035		17LM20001			
Sample Date								12/11/2012		12/15/2013		12/15/2013		12/20/2014		12/20/2014		12/21/2015		12/20/2007			
QC Type								SA		SA		SA		SA		SA		SA		SA			
Study ID								LTM		LTM		LTM		LTM		LTM		LTM		LTM			
Sample Round								5		6		6		7		7		8		1			
Filtered								Total		Dissolved		Total		Dissolved		Total		Total		Total			
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual		
Inorganics																							
Aluminum	UG/L	19,600				25	65	50	UJ	23	UJ	50	UJ	23	U	50	U	18	J	110	J		
Antimony	UG/L	4.4	GA	3	6	16	65	2.7	J	2.3	UJ	2	UJ	2.3	U	2	U	0.5	U	**3.44**			
Arsenic	UG/L	7.8	MCL	10	0	2	65	1.3	UJ	1.3	J	1.3	UJ	1.3	U	1.3	U	1.5	U	4.2	U		
Barium	UG/L	251	GA	1,000	0	65	65	28	J	60	J	56	J	44		41		70		58.8			
Beryllium	UG/L	1.2	MCL	4	0	1	65	0.15	UJ	0.25	UJ	0.15	UJ	0.25	U	0.15	U	0.17	U	0.27	U		
Cadmium	UG/L	1.7	GA	5	0	4	65	0.44	J	0.095	UJ	0.13	UJ	0.095	U	0.13	U	0.15	U	0.36	U		
Calcium	UG/L	195,000				65	65	55,000	J	120,000	J	91,000	J	81,000		77,000		98,000		110,000	J		
Chromium	UG/L	37.2	GA	50	0	4	65	2.5	UJ	2.5	UJ	2.5	UJ	2.5	U	2.5	U	1.6	U	0.84	U		
Cobalt	UG/L	10.5				43	65	0.37	J	0.34	J	0.29	J	0.19	J	0.16	J	0.3	J	0.89	U		
Copper	UG/L	46.7	GA	200	0	33	65	5.4	J	1.1	UJ	1.1	UJ	3.5	J	3.6	J	1.7	U	6.2	J		
Iron	UG/L	25,500	GA	300	15	51	65	90	J	**800**	**J**	**680**	**J**	190		79	J	**360**		140			
Iron+Manganese	UG/L	25,929	GA	500	13	56	60	98.1	J	**897**	**J**	**765**	**J**	199.6		87.7		449		160			
Lead	UG/L	103	MCL	15	1	11	65	1.1	J	0.2	UJ	0.5	UJ	0.23	J	0.5	U	0.98	U	2.9	U		
Magnesium	UG/L	27,300				62	62	7,700	J	24,000	J+	19,000	J	14,000		13,000		19,000		11,000	R		
Manganese	UG/L	911	GA	300	2	60	65	8.1	J	97	J	85	J	9.6		8.7		89		20.5			
Mercury	UG/L	0.14	GA	0.7	0	2	65	0.091	UJ	0.091	UJ	0.091	UJ	0.091	U	0.091	U	0.08	U	0.12	U		
Nickel	UG/L	34	GA	100	0	22	65	2	UJ	2	UJ	2	UJ	2.5	J	2	U	1.9	U	1.2	U		
Potassium	UG/L	7,810				59	60	410	J	500	J	400	J	280	J	330	U	520	J	1,690	R		
Selenium	UG/L	0	GA	10	0	0	65	1.1	UJ	1	UJ	1.1	UJ	1	U	1.1	U	1	U	6.1	U		
Silver	UG/L	0	GA	50	0	0	65	0.18	UJ	0.25	UJ	0.18	UJ	0.25	U	0.18	U	0.1	U	1	U		
Sodium	UG/L	366,000	GA	20,000	4	61	61	2,500	J	6,000	J	4,800	J	3,700		3,500		6,400		6,620	R		
Thallium	UG/L	0.08	MCL	2	0	2	65	0.25	UJ	0.5	UJ	0.25	UJ	0.5	U	0.25	U	0.49	U	0.03	U		
Vanadium	UG/L	32.8				2	65	3.2	UJ	3.8	UJ	3.2	UJ	3.8	U	3.2	U	5.3	U	0.78	U		
Zinc	UG/L	935				35	65	8.4	UJ	8.3	UJ	8.4	UJ	12	J	9	J	9.6	U	72	J		

Notes:

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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17	
Loc ID								MW17-2		MW17-2		MW17-2		MW17-2		MW17-2		MW17-2	
Matrix								GW		GW		GW		GW		GW		GW	
Sample ID								17LM20006		17LM20011FIL		17LM20011UNFIL		17LM20015FIL		17LM20015UNF		17LM20021F	17LM20021U
Sample Date								12/10/2008		11/17/2009		11/17/2009		12/16/2010		12/16/2010		12/11/2012	12/11/2012
QC Type								SA		SA		SA		SA		SA		SA	SA
Study ID								LTM		LTM		LTM		LTM		LTM		LTM	LTM
Sample Round								2		3		3		4		4		5	5
Filtered								Total		Dissolved		Total		Dissolved		Total		Dissolved	Total
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																					
Aluminum	UG/L	19,600				25	65	142	J	88	J	19,600		23	U	51	J	23	UJ	50	UJ
Antimony	UG/L	4.4	GA	3	6	16	65	2.76		2.2		**3.7**		2.3	U	2	U	**4**	**J**	**4.4**	**J**
Arsenic	UG/L	7.8	MCL	10	0	2	65	3.7	U	3.7	U	7.8	J	1.3	U	1.3	U	1.3	UJ	1.3	UJ
Barium	UG/L	251	GA	1,000	0	65	65	51.8		82.3		251		54		58	J	69	J	68	J
Beryllium	UG/L	1.2	MCL	4	0	1	65	0.33	U	0.3	U	1.2	J	0.25	U	0.15	U	0.25	UJ	0.15	UJ
Cadmium	UG/L	1.7	GA	5	0	4	65	0.33	U	0.3	U	1.7		0.095	U	0.13	U	0.095	UJ	0.13	UJ
Calcium	UG/L	195,000				65	65	112,000		154,000	J	195,000	J	140,000		150,000		120,000	J	120,000	J
Chromium	UG/L	37.2	GA	50	0	4	65	2.9	J	0.9	U	37.2		2.5	U	2.5	U	2.5	UJ	2.5	UJ
Cobalt	UG/L	10.5				43	65	1.1	U	1.1	U	10.5		0.32	J	0.46	J	0.39	J	0.42	J
Copper	UG/L	46.7	GA	200	0	33	65	4.4	J	2.9	J	46.7		1.5	J	1.9	J	7.7	J	7.8	J
Iron	UG/L	25,500	GA	300	15	51	65	115		19	UJ	**25,500**	**J**	33	U	130	J	33	UJ	44	UJ
Iron+Manganese	UG/L	25,929	GA	500	13	56	60	121		1.5	J	**25,929**	**J**	23		173		12	J	14	J
Lead	UG/L	103	MCL	15	1	11	65	2.9	U	2.9	U	**103**		0.2	U	0.6	J	0.2	UJ	0.99	J
Magnesium	UG/L	27,300				62	62	11,200		18,200		23,300		18,000		19,000	J	12,000	J	12,000	J
Manganese	UG/L	911	GA	300	2	60	65	6.1		1.5	J	**429**		23		43		12	J	14	J
Mercury	UG/L	0.14	GA	0.7	0	2	65	0.12	U	0.1	U	0.1	U	0.091	U	0.091	U	0.091	UJ	0.091	UJ
Nickel	UG/L	34	GA	100	0	22	65	2.8	J	1.2	J	34		2	U	2	U	2	UJ	2	UJ
Potassium	UG/L	7,810				59	60	1,260	J	2,390		7,810		1,300	J	1,300		2,500	J	2,500	J
Selenium	UG/L	0	GA	10	0	0	65	6.1	U	6.1	U	6.1	U	1	U	1.1	U	1	UJ	1.1	UJ
Silver	UG/L	0	GA	50	0	0	65	1.3	U	1.3	U	1.3	U	0.25	U	0.18	U	0.25	UJ	0.18	UJ
Sodium	UG/L	366,000	GA	20,000	4	61	61	7,860		19,800	J	**20,300**	**J**	14,000	J	14,000	J	8,400	J	8,400	J
Thallium	UG/L	0.08	MCL	2	0	2	65	0.09	U	0.008	U	0.2	U	0.5	U	0.25	U	0.5	UJ	0.25	UJ
Vanadium	UG/L	32.8				2	65	0.98	U	1	U	32.8		3.8	U	3.2	U	3.8	UJ	3.2	UJ
Zinc	UG/L	935				35	65	27.6		28.6		935		17	J	21		24	J	26	J

Notes:

1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17	
Loc ID								MW17-2		MW17-2		MW17-2		MW17-2		MW17-2		MW17-3	
Matrix								GW		GW		GW		GW		GW		GW	
Sample ID								17LM20026F		17LM20026U		17LM20031F		17LM20031U		17LM20036		17LM20002		17LM20007	
Sample Date								12/15/2013		12/15/2013		12/20/2014		12/20/2014		12/20/2015		12/20/2007		12/10/2008	
QC Type								SA		SA		SA		SA		SA		SA		SA	
Study ID								LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round								6		6		7		7		8		1		2	
Filtered								Dissolved		Total		Dissolved		Total		Total		Total		Total	
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																					
Aluminum	UG/L	19,600				25	65	23	UJ	50	UJ	23	U	50	U	19	J	106	J	386	
Antimony	UG/L	4.4	GA	3	6	16	65	2.3	UJ	2	UJ	**3.2**	**J**	**3.3**	**J**	0.63	J	1	U	1	U
Arsenic	UG/L	7.8	MCL	10	0	2	65	1.3	UJ	1.3	UJ	1.3	U	1.3	U	1.5	U	4.2	U	3.7	U
Barium	UG/L	251	GA	1,000	0	65	65	46	J	47	J	63		57		66		39		29.3	
Beryllium	UG/L	1.2	MCL	4	0	1	65	0.25	UJ	0.15	UJ	0.25	U	0.15	U	0.17	U	0.27	U	0.33	U
Cadmium	UG/L	1.7	GA	5	0	4	65	0.095	UJ	0.13	UJ	0.12	J	0.14	J	0.15	U	0.36	U	0.33	U
Calcium	UG/L	195,000				65	65	180,000	J	150,000	J	130,000		120,000		160,000		69,000	J	67,200	
Chromium	UG/L	37.2	GA	50	0	4	65	2.5	UJ	2.5	UJ	2.5	U	2.5	U	1.6	U	0.84	U	0.88	U
Cobalt	UG/L	10.5				43	65	0.44	J	0.38	J	0.15	U	0.13	J	0.42	J	0.89	U	1.1	U
Copper	UG/L	46.7	GA	200	0	33	65	1.1	UJ	1.1	UJ	6.4		6.3		2.4	J	2.6	J	2.8	J
Iron	UG/L	25,500	GA	300	15	51	65	**520**	**J**	**470**	**J**	33	U	46	J	140		133		**1,300**	
Iron+Manganese	UG/L	25,929	GA	500	13	56	60	**594**	**J**	**534**	**J**	2	J	50.1	J	175		170		**1,573**	
Lead	UG/L	103	MCL	15	1	11	65	0.2	UJ	0.5	UJ	0.2	U	0.5	U	0.98	U	2.9	U	2.9	U
Magnesium	UG/L	27,300				62	62	24,000	J+	22,000	J	13,000		11,000		16,000		7,560	R	7,400	
Manganese	UG/L	911	GA	300	2	60	65	74	J	64	J	2	J	4.1	J	35		36.7		273	
Mercury	UG/L	0.14	GA	0.7	0	2	65	0.091	UJ	0.091	UJ	0.091	U	0.091	U	0.08	U	0.12	U	0.12	U
Nickel	UG/L	34	GA	100	0	22	65	2	UJ	2	UJ	2	U	2.2	J	1.9	U	1.2	U	1.8	J
Potassium	UG/L	7,810				59	60	1,100	J	1,000	J	1,600		1,600		1,600		2,620	R	1,840	J
Selenium	UG/L	0	GA	10	0	0	65	1	UJ	1.1	UJ	1	U	1.1	U	1	U	6.1	U	6.1	U
Silver	UG/L	0	GA	50	0	0	65	0.25	UJ	0.18	UJ	0.25	U	0.18	U	0.1	U	1	U	1.3	U
Sodium	UG/L	366,000	GA	20,000	4	61	61	16,000	J	14,000	J	8,800		7,800		12,000		4,550	R	5,500	
Thallium	UG/L	0.08	MCL	2	0	2	65	0.5	UJ	0.25	UJ	0.5	U	0.25	U	0.49	U	0.03	U	0.09	U
Vanadium	UG/L	32.8				2	65	3.8	UJ	3.2	UJ	3.8	U	3.2	U	5.3	U	0.78	U	0.98	U
Zinc	UG/L	935				35	65	11	J	9.3	J	28	J	40	J	26		27	J	14.2	

Notes:

1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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   R = the result was rejected due to QA/QC considerations
   UJ = detection limit is estimated.
3. Shading indicates a concentration above the identified criteria value.
   SA = Sample
   DU = Duplicate Sample
4. Rejected values are not included in the number of samples analyzed.

Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17	
Loc ID								MW17-3		MW17-3		MW17-3		MW17-3		MW17-3		MW17-3	
Matrix								GW		GW		GW		GW		GW		GW	
Sample ID								17LM20012FIL		17LM20012UNFIL		17LM20017FIL		17LM20017UNF		17LM20022F		17LM20027F	
Sample Date								11/18/2009		11/18/2009		12/16/2010		12/16/2010		12/11/2012		12/15/2013	
QC Type								SA		SA		SA		SA		SA		SA	
Study ID								LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round								3		3		4		4		5		6	
Filtered								Dissolved		Total		Dissolved		Total		Dissolved		Dissolved	
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																			
Aluminum	UG/L	19,600				25	65	141	J	1,550	J	23	U	50	U	23	UJ	23	UJ
Antimony	UG/L	4.4	GA	3	6	16	65	1	U	1.5		2.3	U	2	U	2.3	UJ	2.3	UJ
Arsenic	UG/L	7.8	MCL	10	0	2	65	3.7	U	3.7	U	1.3	U	1.3	U	1.3	UJ	1.3	UJ
Barium	UG/L	251	GA	1,000	0	65	65	49.4		54.5		37		38	J	37	J	52	J
Beryllium	UG/L	1.2	MCL	4	0	1	65	0.3	U	0.3	U	0.25	U	0.15	U	0.25	UJ	0.25	UJ
Cadmium	UG/L	1.7	GA	5	0	4	65	0.3	U	0.3	U	0.095	U	0.13	U	0.095	UJ	0.095	UJ
Calcium	UG/L	195,000				65	65	99,400	J	95,900	J	90,000		93,000		74,000	J	130,000	J
Chromium	UG/L	37.2	GA	50	0	4	65	0.9	U	5.2		2.5	U	2.5	U	2.5	UJ	2.5	UJ
Cobalt	UG/L	10.5				43	65	1.5	J	1.7	J	0.63		0.7		0.15	UJ	0.31	J
Copper	UG/L	46.7	GA	200	0	33	65	2.5	J	7.9	J	1.1	U	1.1	U	3.3	J	1.3	J
Iron	UG/L	25,500	GA	300	15	51	65	**827**	**J**	**2,690**	**J**	**730**	**J**	**770**	**J**	33	UJ	33	UJ
Iron+Manganese	UG/L	25,929	GA	500	13	56	60	**968**	**J**	**2,858**	**J**	**890**		**940**		34	U	2.3	J
Lead	UG/L	103	MCL	15	1	11	65	2.9	U	8.6		0.2	U	0.5	U	0.24	J	0.35	J
Magnesium	UG/L	27,300				62	62	9,850		9,170		9,900		10,000	J	6,100	J	15,000	J+
Manganese	UG/L	911	GA	300	2	60	65	141		168		160		170		1	UJ	2.3	J
Mercury	UG/L	0.14	GA	0.7	0	2	65	0.1	U	0.1	U	0.091	U	0.091	U	0.091	UJ	0.091	UJ
Nickel	UG/L	34	GA	100	0	22	65	3.1	J	4.5	J	2	U	2	U	2	UJ	2	UJ
Potassium	UG/L	7,810				59	60	1,290		1,590		1,200	J	1,200		1,800	J	870	J
Selenium	UG/L	0	GA	10	0	0	65	6.1	U	6.1	U	1	U	1.1	U	1	UJ	1	UJ
Silver	UG/L	0	GA	50	0	0	65	1.3	U	1.3	U	0.25	U	0.18	U	0.25	UJ	0.25	UJ
Sodium	UG/L	366,000	GA	20,000	4	61	61	7,500	J	6,200	J	6,000	J	6,100	J	3,300	J	11,000	J
Thallium	UG/L	0.08	MCL	2	0	2	65	0.008	U	0.008	U	0.5	U	0.25	U	0.5	UJ	0.5	UJ
Vanadium	UG/L	32.8				2	65	1	U	1.7	J	3.8	U	3.2	U	3.8	UJ	3.8	UJ
Zinc	UG/L	935				35	65	21.1		45.7		8.3	U	12	J	29	J	26	J	35	J

Notes:
1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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3. Shading indicates a concentration above the identified criteria value.
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   DU = Duplicate Sample
4. Rejected values are not included in the number of samples analyzed.

Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17	
Loc ID								MW17-3		MW17-3		MW17-3		MW17-3		MW17-4		MW17-4	
Matrix								GW		GW		GW		GW		GW		GW	
Sample ID								17LM20027U		17LM20032F		17LM20032U		17LM20037		17LM20003		17LM20008		17LM20013FIL	
Sample Date								12/15/2013		12/20/2014		12/20/2014		12/20/2015		12/20/2007		12/10/2008		11/17/2009	
QC Type								SA		SA		SA		SA		SA		SA		SA	
Study ID								LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round								6		7		7		8		1		2		3	
Filtered								Total		Dissolved		Total		Total		Total		Total		Dissolved	
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																					
Aluminum	UG/L	19,600				25	65	50	UJ	23	U	180		31	J	50.2	J	125	J	28	J
Antimony	UG/L	4.4	GA	3	6	16	65	2	UJ	2.3	U	2	U	0.5	U	1	U	0.62	J	1	U
Arsenic	UG/L	7.8	MCL	10	0	2	65	1.3	UJ	1.3	U	1.3	U	1.5	U	4.2	U	3.7	U	3.7	U
Barium	UG/L	251	GA	1,000	0	65	65	53	J	41		38		51		32.5		35.9		36.3	
Beryllium	UG/L	1.2	MCL	4	0	1	65	0.15	UJ	0.25	U	0.15	U	0.17	U	0.27	U	0.33	U	0.3	U
Cadmium	UG/L	1.7	GA	5	0	4	65	0.13	UJ	0.095	U	0.13	U	0.15	U	0.36	U	0.33	U	0.3	U
Calcium	UG/L	195,000				65	65	110,000	J	73,000		69,000		100,000		74,900	J	74,700		96,600	J
Chromium	UG/L	37.2	GA	50	0	4	65	2.5	UJ	2.5	U	2.5	U	1.6	U	1	J	0.88	U	0.9	U
Cobalt	UG/L	10.5				43	65	0.3	J	0.15	U	0.12	J	0.12	U	0.89	U	2.4	J	1.5	J
Copper	UG/L	46.7	GA	200	0	33	65	1.1	J	13		15		1.7	U	1.8	J	1.8	J	1.3	U
Iron	UG/L	25,500	GA	300	15	51	65	110	J	33	U	160		43	J	45.4	J	**1,760**		60	J
Iron+Manganese	UG/L	25,929	GA	500	13	56	60	112	J	5.1		166.1		44.8	J	59	J	**2,671**		258	J
Lead	UG/L	103	MCL	15	1	11	65	0.5	UJ	0.2	U	1.1	J	0.98	U	2.9	U	2.9	U	2.9	U
Magnesium	UG/L	27,300				62	62	15,000	J	5,800		5,600		11,000		10,400	R	10,200		12,900	
Manganese	UG/L	911	GA	300	2	60	65	2	J	5.1		6.1		1.8	U	13.7		**911**		198	
Mercury	UG/L	0.14	GA	0.7	0	2	65	0.091	UJ	0.091	U	0.091	U	0.08	U	0.12	U	0.12	U	0.1	U
Nickel	UG/L	34	GA	100	0	22	65	2	UJ	2.6	J	2	J	1.9	U	1.2	U	2.6	J	2.2	J
Potassium	UG/L	7,810				59	60	840	J	1,400		1,500		810	J	838	R	1,190	J	844	
Selenium	UG/L	0	GA	10	0	0	65	1.1	UJ	1	U	1.1	U	1	U	6.1	U	6.1	U	6.1	U
Silver	UG/L	0	GA	50	0	0	65	0.18	UJ	0.25	U	0.18	U	0.1	U	1	U	1.3	U	1.3	U
Sodium	UG/L	366,000	GA	20,000	4	61	61	10,000	J	1,900		1,900		8,400		**28,500**	**J**	15,500		10,400	J
Thallium	UG/L	0.08	MCL	2	0	2	65	0.25	UJ	0.5	U	0.25	U	0.49	U	0.03	U	0.09	U	0.008	U
Vanadium	UG/L	32.8				2	65	3.2	UJ	3.8	U	3.2	U	5.3	U	0.78	U	0.98	U	1	U
Zinc	UG/L	935				35	65	33	J	42	J	44	J	27		5.1	J	6.7	J	3.6	U

Notes:

1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17	
Loc ID								MW17-4		MW17-4		MW17-4		MW17-4		MW17-4		MW17-4	
Matrix								GW		GW		GW		GW		GW		GW	
Sample ID								17LM20013UNFIL		17LM20018FIL		17LM20018UNF		17LM20023F		17LM20023U		17LM20028F		17LM20028U	
Sample Date								11/17/2009		12/16/2010		12/16/2010		12/11/2012		12/11/2012		12/15/2013		12/15/2013	
QC Type								SA		SA		SA		SA		SA		SA		SA	
Study ID								LTM		LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round								3		4		4		5		5		6		6	
Filtered								Total		Dissolved		Total		Dissolved		Total		Dissolved		Total	
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																					
Aluminum	UG/L	19,600				25	65	70	J	23	U	50	U	23	UJ	50	UJ	23	UJ	50	UJ
Antimony	UG/L	4.4	GA	3	6	16	65	1	U	2.3	U	2	U	2.3	UJ	2	UJ	2.3	UJ	2	UJ
Arsenic	UG/L	7.8	MCL	10	0	2	65	3.7	U	1.3	U	1.3	U	1.3	UJ	1.3	UJ	1.3	UJ	1.3	UJ
Barium	UG/L	251	GA	1,000	0	65	65	36.6		27		28	J	65	J	67	J	20	J	23	J
Beryllium	UG/L	1.2	MCL	4	0	1	65	0.3	U	0.25	U	0.15	U	0.25	UJ	0.15	UJ	0.25	UJ	0.15	UJ
Cadmium	UG/L	1.7	GA	5	0	4	65	0.3	U	0.095	U	0.13	U	0.095	UJ	0.13	UJ	0.095	UJ	0.13	UJ
Calcium	UG/L	195,000				65	65	97,600	J	90,000		88,000		83,000	J	87,000	J	96,000	J	93,000	J
Chromium	UG/L	37.2	GA	50	0	4	65	0.9	U	2.5	U	2.5	U	2.5	UJ	2.5	UJ	2.5	UJ	2.5	UJ
Cobalt	UG/L	10.5				43	65	1.3	J	0.96		1.1		0.21	J	0.25	J	1	J	1.1	J
Copper	UG/L	46.7	GA	200	0	33	65	1.3	U	1.1	U	1.1	U	1.1	J	5	UJ	1.1	UJ	1.1	UJ
Iron	UG/L	25,500	GA	300	15	51	65	142	J	240	J	260	J	33	UJ	72	J	**810**	**J**	**810**	**J**
Iron+Manganese	UG/L	25,929	GA	500	13	56	60	355	J	370		400		9.5	J	83	J	**1,090**	**J**	**1,090**	**J**
Lead	UG/L	103	MCL	15	1	11	65	2.9	U	0.2	U	0.5	U	0.2	UJ	0.5	UJ	0.2	UJ	0.5	UJ
Magnesium	UG/L	27,300				62	62	13,000		13,000		13,000	J	15,000	J	15,000	J	15,000	J+	15,000	J
Manganese	UG/L	911	GA	300	2	60	65	213		130		140		9.5	J	11	J	280	J	280	J
Mercury	UG/L	0.14	GA	0.7	0	2	65	0.1	U	0.091	U	0.091	U	0.091	UJ	0.091	UJ	0.091	UJ	0.091	UJ
Nickel	UG/L	34	GA	100	0	22	65	2.4	J	2	U	2	U	2	UJ	2.1	J	2	UJ	2	UJ
Potassium	UG/L	7,810				59	60	866		540		530	J	750	J	780	J	450	J	430	J
Selenium	UG/L	0	GA	10	0	0	65	6.1	U	1	U	1.1	U	1	UJ	1.1	UJ	1	UJ	1.1	UJ
Silver	UG/L	0	GA	50	0	0	65	1.3	U	0.25	U	0.18	U	0.25	UJ	0.18	UJ	0.25	UJ	0.18	UJ
Sodium	UG/L	366,000	GA	20,000	4	61	61	10,500	J	12,000	J	12,000	J	8,900	J	8,600	J	7,800	J	7,800	J
Thallium	UG/L	0.08	MCL	2	0	2	65	0.008	U	0.5	U	0.25	U	0.5	UJ	0.25	UJ	0.5	UJ	0.25	UJ
Vanadium	UG/L	32.8				2	65	1	U	3.8	U	3.2	U	3.8	UJ	3.2	UJ	3.8	UJ	3.2	UJ
Zinc	UG/L	935				35	65	3.6	U	8.7	J	8.4	U	8.3	UJ	8.4	UJ	8.3	UJ	8.4	UJ

Notes:

1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17	
Loc ID								MW17-4		MW17-4		MW17-4		MW17-5		MW17-5		MW17-5	
Matrix								GW		GW		GW		GW		GW		GW	
Sample ID								17LM20033F		17LM20033U		17LM20038		17LM20004		17LM20009		17LM20014FIL	17LM20014UNFIL
Sample Date								12/20/2014		12/20/2014		12/21/2015		12/20/2007		12/11/2008		11/17/2009	11/17/2009
QC Type								SA		SA		SA		SA		SA		SA	SA
Study ID								LTM		LTM		LTM		LTM		LTM		LTM	LTM
Sample Round								7		7		8		1		2		3	3
Filtered								Dissolved		Total		Total		Total		Total		Dissolved	Total
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																					
Aluminum	UG/L	19,600				25	65	23	U	50	U	18	U	98.5	J	125	J	29	J	98	J
Antimony	UG/L	4.4	GA	3	6	16	65	2.3	U	2	U	0.56	J	1	U	0.56	J	1		1	
Arsenic	UG/L	7.8	MCL	10	0	2	65	1.3	U	1.3	U	1.5	U	4.2	U	3.7	U	3.7	U	3.7	U
Barium	UG/L	251	GA	1,000	0	65	65	27		27		29		86.7		82.9		166		168	
Beryllium	UG/L	1.2	MCL	4	0	1	65	0.25	U	0.15	U	0.17	U	0.27	U	0.33	U	2	U	2	U
Cadmium	UG/L	1.7	GA	5	0	4	65	0.095	U	0.13	U	0.15	U	0.36	U	0.33	U	0.3	U	0.3	U
Calcium	UG/L	195,000				65	65	80,000		75,000		80,000		97,100	J	97,300		184,000	J	185,000	J
Chromium	UG/L	37.2	GA	50	0	4	65	2.5	U	2.5	U	1.6	U	0.84	U	0.88	U	0.9	U	0.9	U
Cobalt	UG/L	10.5				43	65	0.31	J	0.24	J	1.1		0.89	U	1.1	U	1.1	U	1.1	U
Copper	UG/L	46.7	GA	200	0	33	65	2.3	J	2.8	J	1.7	U	1.3	U	1.5	J	1.3	U	1.3	U
Iron	UG/L	25,500	GA	300	15	51	65	120		130		59	J	91.7		76		19	UJ	34	J
Iron+Manganese	UG/L	25,929	GA	500	13	56	60	260		250		158	J	128		85		24.3		61.4	J
Lead	UG/L	103	MCL	15	1	11	65	0.2	U	0.5	U	1.5	J	2.9	U	2.9	U	2.9	U	2.9	U
Magnesium	UG/L	27,300				62	62	12,000		11,000		11,000		15,800	J	15,600		27,100		27,300	
Manganese	UG/L	911	GA	300	2	60	65	140		120		99		36.5		8.9		24.3		27.4	
Mercury	UG/L	0.14	GA	0.7	0	2	65	0.091	U	0.091	U	0.08	U	0.12	U	0.12	U	0.1	U	0.1	U
Nickel	UG/L	34	GA	100	0	22	65	3	J	2	J	2.1	J	1.2	U	1.2	J	1.7	J	1.8	J
Potassium	UG/L	7,810				59	60	480	J	420	J	500	J	972	R	824	J	1,920		1,960	
Selenium	UG/L	0	GA	10	0	0	65	1	U	1.1	U	1	U	6.1	U	6.1	U	6.1	U	6.1	U
Silver	UG/L	0	GA	50	0	0	65	0.25	U	0.18	U	0.1	U	1	U	1.3	U	1.3	U	1.3	U
Sodium	UG/L	366,000	GA	20,000	4	61	61	7,700		7,300		6,000		7,950	R	7,360		**364,000**	**J**	**366,000**	**J**
Thallium	UG/L	0.08	MCL	2	0	2	65	0.5	U	0.25	U	0.49	U	0.03	U	0.09	U	0.08	J	0.08	J
Vanadium	UG/L	32.8				2	65	3.8	U	3.2	U	5.3	U	0.78	U	0.98	U	1	U	1	U
Zinc	UG/L	935				35	65	8.3	U	8.4	U	9.6	U	4.7	J	41.6		3.6	U	3.6	U

Notes:

1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area							SEAD-17		SEAD-17		SEAD-17		SEAD-17		SEAD-17	
Loc ID							MW17-5		MW17-5		MW17-5		MW17-5		MW17-5	
Matrix							GW		GW		GW		GW		GW	
Sample ID							17LM20019FIL		17LM20019UNF		17LM20024F		17LM20024U		17LM20029F		17LM20029U	
Sample Date							12/16/2010		12/16/2010		12/11/2012		12/11/2012		12/15/2013		12/15/2013	
QC Type							SA		SA		SA		SA		SA		SA	
Study ID							LTM		LTM		LTM		LTM		LTM		LTM	
Sample Round							4		4		5		5		6		6	
Filtered							Dissolved		Total		Dissolved		Total		Dissolved		Total	
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual	Value	Qual
Inorganics																			
Aluminum	UG/L	19,600				25	65	23	U	50	U	23	UJ	50	UJ	23	UJ	50	UJ
Antimony	UG/L	4.4	GA	3	6	16	65	2.3	U	2	U	2.3	UJ	2	UJ	2.3	UJ	2	UJ
Arsenic	UG/L	7.8	MCL	10	0	2	65	1.3	U	1.3	U	1.3	UJ	1.3	UJ	1.3	UJ	1.3	UJ
Barium	UG/L	251	GA	1,000	0	65	65	81		82	J	24	J	26	J	75	J	86	J
Beryllium	UG/L	1.2	MCL	4	0	1	65	0.25	U	0.15	U	0.25	UJ	0.15	UJ	0.25	UJ	0.15	UJ
Cadmium	UG/L	1.7	GA	5	0	4	65	0.095	U	0.13	U	0.095	UJ	0.13	UJ	0.095	UJ	0.13	UJ
Calcium	UG/L	195,000				65	65	100,000		110,000		68,000	J	75,000	J	110,000	J	100,000	J
Chromium	UG/L	37.2	GA	50	0	4	65	2.5	U	2.5	U	2.5	UJ	2.5	UJ	2.5	UJ	2.5	UJ
Cobalt	UG/L	10.5				43	65	0.17	J	0.19	J	0.31	J	0.31	J	0.2	J	0.22	J
Copper	UG/L	46.7	GA	200	0	33	65	1.1	U	1.1	U	3.7	J	5	UJ	1.1	UJ	1.1	UJ
Iron	UG/L	25,500	GA	300	15	51	65	83	J	110	J	44	J	160	J	**350**	**J**	140	J
Iron+Manganese	UG/L	25,929	GA	500	13	56	60	118		145		82	J	219	J	374	J	167	J
Lead	UG/L	103	MCL	15	1	11	65	0.2	U	0.5	U	0.2	UJ	0.5	UJ	0.2	UJ	0.5	UJ
Magnesium	UG/L	27,300				62	62	17,000		18,000	J	9,900	J	11,000	J	18,000	J+	17,000	J
Manganese	UG/L	911	GA	300	2	60	65	35		35		38	J	59	J	24	J	27	J
Mercury	UG/L	0.14	GA	0.7	0	2	65	0.091	U	0.091	U	0.12	J	0.091	UJ	0.091	UJ	0.091	UJ
Nickel	UG/L	34	GA	100	0	22	65	2	U	2	U	2	UJ	2	UJ	2	UJ	2	UJ
Potassium	UG/L	7,810				59	60	1,600	J	1,600		460	J	460	J	1,200	J	1,100	J
Selenium	UG/L	0	GA	10	0	0	65	1	U	1.1	U	1	UJ	1.1	UJ	1	UJ	1.1	UJ
Silver	UG/L	0	GA	50	0	0	65	0.25	U	0.18	U	0.25	UJ	0.18	UJ	0.25	UJ	0.18	UJ
Sodium	UG/L	366,000	GA	20,000	4	61	61	8,200	J	8,300	J	9,400	J	9,100	J	5,400	J	5,300	J
Thallium	UG/L	0.08	MCL	2	0	2	65	0.5	U	0.25	U	0.5	UJ	0.25	UJ	0.5	UJ	0.25	UJ
Vanadium	UG/L	32.8				2	65	3.8	U	3.2	U	3.8	UJ	3.2	UJ	3.8	UJ	3.2	UJ
Zinc	UG/L	935				35	65	20		8.4	U	8.3	UJ	8.4	UJ	8.3	UJ	8.4	UJ

Notes:

1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
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3. Shading indicates a concentration above the identified criteria value.
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Appendix D
Post-Remedial Action Groundwater Monitoring Results (Years 1 through 8)
Annual Report - SEAD 16 and SEAD 17
Seneca Army Depot Activity

Area								SEAD-17		SEAD-17		SEAD-17		
Loc ID								MW17-5		MW17-5		MW17-5		
Matrix								GW		GW		GW		
Sample ID								17LM20034F		17LM20034U		17LM20039		
Sample Date								12/20/2014		12/20/2014		12/20/2015		
QC Type								SA		SA		SA		
Study ID								LTM		LTM		LTM		
Sample Round								7		7		8		
Filtered								Dissolved		Total		Total		
Parameter	Unit	Maximum Value	Criteria Source	Criteria Level	Number of Exceedances	Number of Times Detected	Number of Samples Analyzed	Value	Qual	Value	Qual	Value	Qual
Inorganics														
Aluminum	UG/L	19,600				25	65	23	U	50	U	18	U
Antimony	UG/L	4.4	GA	3	6	16	65	2.3	U	2	U	0.5	U
Arsenic	UG/L	7.8	MCL	10	0	2	65	1.3	U	1.3	U	1.5	U
Barium	UG/L	251	GA	1,000	0	65	65	83		92		86	
Beryllium	UG/L	1.2	MCL	4	0	1	65	0.25	U	0.15	U	0.17	U
Cadmium	UG/L	1.7	GA	5	0	4	65	0.095	U	0.13	U	0.15	U
Calcium	UG/L	195,000				65	65	91,000		100,000		100,000	
Chromium	UG/L	37.2	GA	50	0	4	65	2.5	U	2.5	U	1.6	U
Cobalt	UG/L	10.5				43	65	0.15	U	0.12	U	0.14	J
Copper	UG/L	46.7	GA	200	0	33	65	1.1	U	2.6	J	1.7	U
Iron	UG/L	25,500	GA	300	15	51	65	33	U	55	J	43	J
Iron+Manganese	UG/L	25,929	GA	500	13	56	60	34	U	46	U	48.8	J
Lead	UG/L	103	MCL	15	1	11	65	0.2	U	0.5	U	0.98	U
Magnesium	UG/L	27,300				62	62	14,000		15,000		17,000	
Manganese	UG/L	911	GA	300	2	60	65	1	U	2	U	5.8	
Mercury	UG/L	0.14	GA	0.7	0	2	65	0.091	U	0.091	U	0.08	U
Nickel	UG/L	34	GA	100	0	22	65	2.8	J	2	U	1.9	U
Potassium	UG/L	7,810				59	60	810		860	J	1,300	
Selenium	UG/L	0	GA	10	0	0	65	1	U	1.1	U	1	U
Silver	UG/L	0	GA	50	0	0	65	0.25	U	0.18	U	0.1	U
Sodium	UG/L	366,000	GA	20,000	4	61	61	4,900		4,900		5,800	
Thallium	UG/L	0.08	MCL	2	0	2	65	0.5	U	0.25	U	0.49	U
Vanadium	UG/L	32.8				2	65	3.8	U	3.2	U	5.3	U
Zinc	UG/L	935				35	65	8.3	U	8.4	U	9.6	U

Notes:

1. The lowest value for either the New York Class GA Groundwater Standards (TOGS 1.1.1, June 1998, et al.) or the EPA Maximum Contaminant Limit (MCL), source http://www.epa.gov/safewater/mcl.html#inorganic.html is used. A blank cell indicates no criteria value available.
2. Data validation qualifier.
   [empty cell] = data is not qualified
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3. Shading indicates a concentration above the identified criteria value.
   SA = Sample
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4. Rejected values are not included in the number of samples analyzed.

APPENDIX E

LABORATORY ANALYTICAL REPORT

Laboratory Reports are provided on the CD version of this report.

APPENDIX F

DATA VALIDATION

PROJECT NAME/NO. USACE - Seneca Army Depot SEAD-16/17 LTM Round 8
LAB: TestAmerica
SDG: 680-120341-1
FRACTION: Metals (SW846 6020A)
MEDIA: Groundwater
NUMBER OF SAMPLES: 12

CRITERIA	Did Analyses Meet all criteria as specified in the SOPS?	Region 2 Acceptable limits / criteria	Comments/Qualifying Actions	Qualifiers Added?
Data Completeness, Holding Times & Preservation	Yes	Cooler temp < 10 C. pH < 2. Holding Time Hg < 28 days, all other metals < 180 days from collection.	Coolers were received at 1.2-1.6°C by the laboratory. All samples were received in good condition based on the laboratory login report. Samples were properly preserved and had pH < 2.	No
Calibration	Yes	r^2 $\geq$ 0.995 CCV every 10 samps or 2 hours ICV/CCV %R btw 90-110%	Calibrations available, taken every ten samples, and within recovery limits (90-110%).	No
Blanks (prep blank, ICB, CCB)	Yes	Method blanks: 1 per 20 project samples.	All ICB, CCB, and laboratory preparation blanks associated with project samples did not contain contamination.	No
CRDL Standard	Yes	CRDL results btw 70-130%	CRDL analyses for all remaining metals conducted at the beginning and end of the analysis. All met requirements.	No
Laboratory Control Sample	Yes	LCS/LCSD: 1 per 20 project samples or each preparation batch. LCS limits within 80-120%.	All LCS recoveries were acceptable and within QC limits.	No
Duplicates	Yes	RPD < 20% or Absolute Diff < RL when samp/dup value < 5x RL	All laboratory duplicate results were acceptable and within QC limits.	No

PROJECT NAME/NO. USACE - Seneca Army Depot SEAD-16/17 LTM Round 8
LAB: TestAmerica
SDG: 680-120341-1
FRACTION: Metals (SW846 6020A)
MEDIA: Groundwater
NUMBER OF SAMPLES: 12

CRITERIA	Did Analyses Meet all criteria as specified in the SOPS?	Region 2 Acceptable limits / criteria	Comments/Qualifying Actions	Qualifiers Added?
Matrix Spike/Matrix Spike Duplicates	No	MS/MSD: 1 per 20 project samples or each preparation batch. Recoveries within lab limits. MS/MSD %RPDs <= 20%. Spike Recovery limits 75-125%	Sample 16LM20054 was designated for MS/MSD analysis. All precision and accuracy results were acceptable with the exception of potassium (-146%R/-140%R) and antimony (-87%R/-96%R). The post digestion spike also experienced low recoveries for potassium (-18%R) and antimony (13%R). Therefore, the positive potassium and antimony results for the parent sample were considered estimated, possibly biased low, and qualified "J-".	Yes
ICP Interference Check Sample (ICS)	Yes	ICS results within 80-120%.	All concentrations detected in all samples within the ICP Linear Range. No action was taken.	No
ICP Tune Analysis	Yes	RSD < 5%	All isotopes of each analyte had a RSD < 5%.	No
Internal Standard	Yes	Intensity within 60-125%	IS had %RI within acceptance limits.	No
Serial Dilution	No	Performed on samples of a similar matrix or 1 per 20 samples. %D ≤ 10% conc ≥ 25xDL (7470A/7471A) and 10x IDL (6010B) for 5-fold dilution.	Serial dilution was conducted on sample 16LM20054 with all results considered compliant with the exception of the serial dilution results for barium (80%D), caclcium (79%D), potassium (78%D), magnesium (80%D), sodium (79%D), and antimony (80%D). Therefore, positive results for these analytes were considered estimated and qualified "J" for the parent sample..	Yes
Total/Dissolved Comparison	Yes	%RPD less than 20%	Samples were collected for total analysis.	No
Field Duplicate Precision	No	%RPD less than 30%	Sample 16LM20055 was collected as the field duplicate sample of 16LM20054. Precision results were considered acceptable with the exception of barium (129%RPD), calcium (129%RPD), potassium (123%RPD), magnesium (132%RPD), manganese (111%RPD), sodium (118%RPD), lead (131%RPD), and antimony (145%RPD). Results for these analytes were considered estimated and qualified "J".	Yes

RT = Retention Time; %D = Percent Deviation; %RPD = Relative Percent Difference; %RSD = Percent Relative Standard Deviation; RRF = Relative Response Factor; CCV = Continuing Calibration Verification
TCL = Target Compound List; MS = Matrix Spike; MSD = Matrix Spike Duplicate;

PROJECT NAME/NO. USACE - Seneca Army Depot SEAD-16/17 LTM Round 8
LAB: TestAmerica
SDG: 680-120341-1
FRACTION: Metals (SW846 7470A)
MEDIA: Groundwater
NUMBER OF SAMPLES: 12

CRITERIA	Did Analyses Meet all criteria as specified in the SOPS?	Region 2 Acceptable limits / criteria	Comments/Qualifying Actions	Qualifiers Added?
Data Completeness, Holding Times & Preservation	Yes	Cooler temp < 10 C. Holding Time Hg < 28 days, all other metals < 180 days from collection.	Coolers were received at 1.2-1.6°C by the laboratory. All samples were received in good condition based on the laboratory login report. Samples were properly preserved and had pH < 2.	No
Calibration	Yes	r^2 ≥ 0.995 CCV every 10 samples or 2 hours ICV/CCV %R btw 80-120% (specific to Hg)	Calibrations available, taken every ten samples, and within recovery limits (80-120%).	No
Blanks (prep blank, ICB, CCB)	Yes	Method blanks: 1 per 20 project samples.	ICB, CCB, and preparation blanks did not contain mercury.	No
CRDL Standard	Yes	CRDL results btw 70-130%	CRDL analyses for Hg conducted at the beginning and end of the analysis. All met requirements.	No
Laboratory Control Sample	Yes	LCS/LCSD: 1 per 20 project samples or each preparation batch. LCS limits within 80-120%.	All LCSs within QC limits.	No
Duplicates	Yes	RPD < 20% or Absolute Diff < RL when samp/dup value < 5x RL	All laboratory duplicate results were within criteria for mercury.	No
Matrix Spike/Matrix Spike Duplicates	Yes	MS/MSD: 1 per 20 project samples or each preparation batch. Recoveries within lab limits. MS/MSD %RPDs <= 20%. Spike Recovery limits 75-125%	Sample 16LM20054 was designated for MS/MSD analysis. Precision and accuracy results for mercury were compliant.	No
ICP Interference Check Sample (ICS)	Yes	ICS results within 80-120%.	ICP Interference Check was performed and all recoveries were within acceptance limits.	No
Serial Dilution	NA	Performed on samples of a similar matrix or 1 per 20 samples. %D ≤ 10% conc ≥ 25xDL (7470A/7471A) and 10x IDL (6010B) for 5-fold dilution.	A serial dilution was not performed on this analysis.	NA
Total/Dissolved Comparison	Yes	%RPD less than 20%	All samples were collected for total analysis.	No
Field Duplicate Precision	Yes	%RPD less than 30%	Sample 16LM20055 was collected as the field duplicate of 16LM20054. Mercury was not detected in either sample.	No

APPENDIX G

RESPONSE TO COMMENTS

Comments on the Draft Year 7 Annual Report were addressed within this Year 8 Annual Report

Army's Response to Comments from the United States Environmental Protection Agency

Subject: Draft Annual Report – Year 7
Abandoned Deactivation Furnace (SEAD-16)
and Active Deactivation Furnace (SEAD-17)
Seneca Army Depot Activity
Romulus, New York

Comments Dated: September 9, 2015

Date of Comment Response: April 27, 2016

Army's Response to Comments

GENERAL COMMENTS

Comment 1: The Annual Report proposes to conclude annual monitoring, but does not provide sufficient justification for why groundwater monitoring is no longer necessary at SEAD-16 and SEAD-17. The following issues should be addressed before considering discontinuing annual groundwater monitoring:

a. Section 5.2, Recommendations, indicates that annual monitoring will be concluded because the 95% upper confidence limit (UCL) for antimony in SEAD-16 and SEAD-17 groundwater is less than the SEAD background average concentration. However, it is unclear why the 95% UCL for each site is used for comparison to background concentrations, considering that data from upgradient, sidegradient and downgradient wells are used to calculate the 95% UCL. Typically, data from individual wells, especially those downgradient of current or former source areas, are used to determine groundwater impacts and potential human exposures. Revise the Annual Report to remove the comparison of the 95% UCL for antimony to the background average concentration, or explain how the 95% UCL is representative and protective of potential drinking water exposures.

b. Elevated concentrations of sodium, in excess of the GA standard by an order of magnitude, are noted at SEAD-16. The Annual Report indicates that the sodium concentrations are possibly from the salt piles located upgradient of the site. However, the Annual Report does not provide further support for this statement. The Annual Report should provide additional information or sample results that show that the sodium contamination is from an offsite source.

Please revise the Annual Report to address these issues or indicate that these issues will be addressed prior to concluding annual monitoring.

Response 1:

a) All comparisons of the 95% UCL for antimony to the background average concentration were removed from both the text and the figures of the Annual Report. Further recommendations regarding the conclusion of sampling at SEAD 16/17 will be presented in the 2016 5-Year Review.

Manganese followed a similar trend as iron at well MW16-5 during Year 7. The manganese (dissolved) result was greater than the total result (160 µg/L and 130 µg/L, respectively). Neither value was above the NYS Class GA standard of 300 µg/L.
During the Year 8 sampling event, no dissolved samples were collected; however, iron concentrations in MW16-5 and MW16-6 were above the NYS Class GA standard, but below the SEDA background concentrations. Refer to Sections 3.1.4 and 3.1.6.1 in the Year 8 Annual Report.

Comment 4: Section 3.1.6, LTM Groundwater Data Trends, discusses trends in groundwater concentrations of iron at SEAD-16 and SEAD-17, but a figure showing these trends is not provided. For example, Section 3.1.6.1, LTM Groundwater Trends for SEAD-16, states that concentrations of iron at SEAD-16 are approaching the NYS Class GA standard. A figure similar to Figures 6A and 6B would be helpful in showing the trends in concentrations of iron measured at site monitoring wells through time. Please revise the Annual Report to include a figure showing the concentrations of iron in the current and previous monitoring events.

Response 4: Two figures were added to the Year 8 Annual Report (Figure 6C and 6D) to show the concentrations of iron during the current and previous monitoring events at SEAD-16 and SEAD-17, respectively.

SPECIFIC COMMENTS

Comment 1: Section 3.1.6.2, LTM Groundwater Trends for SEAD-17, Page 3-6: The text appears to correlate sodium concentrations that exceed the groundwater standard to regional iron and manganese concentrations, but further discussion to support this correlation is not provided. This section states, "Many of the identified groundwater quality exceedances of sodium appeared either as random occurrences (e.g., sodium at MW17-5 in Year 3) or may be attributable to iron and manganese groundwater concentrations that are identified regionally in Seneca County and consistent with the Seneca groundwater background levels presented in Appendix B." Please revise the text to clarify how the sodium exceedances are correlated to the iron and manganese concentrations in groundwater.

Response 1: The quoted text in the RTC was removed and was replaced with the following:

> There are no trends associated with the elevated concentrations of sodium at SEAD-17 (Appendix D). These concentrations are estimated and, in general, return to the historical baseline condition at each well. Typically, sodium concentrations at SEAD-17 are below the Seneca background (Appendix B).

Comment 2: Section 5.2, Recommendations, Page 5-1: The recommendation for continued land use control (LUC) inspections indicates they will be performed periodically, but it is unclear at what frequency they will be performed. Please revise this section to specify the frequency at which LUC inspections will be performed.

Response 2: The LUC inspections will continue to be performed on an annual basis. The text was revised accordingly.

b) The following text was added to Section 3.1.6.1:

> As identified on **Figure 5**, the groundwater east (upgradient) of SEAD-16 travels towards the southwest, from the salt pile storage area towards SEAD-16. In satellite photos of the area, the "Unnamed Dirt Road" that originates from the salt storage area and extends towards SEAD-16 appears to have a white coloration; the white coloration is likely due to salt residue from runoff emanating from the salt pile.
>
> Historically, the highest concentrations of sodium were found in well MW16-4; this well is the most directly in line with the suspected path of the salt. The location of the Seneca County Highway Department salt pile storage area is indicated on **Figure 5**. Sampling has not been conducted at the salt pile, or immediately downgradient of it, as it is not a CERCLA release; the Army does not plan on conducting any sampling in this location.

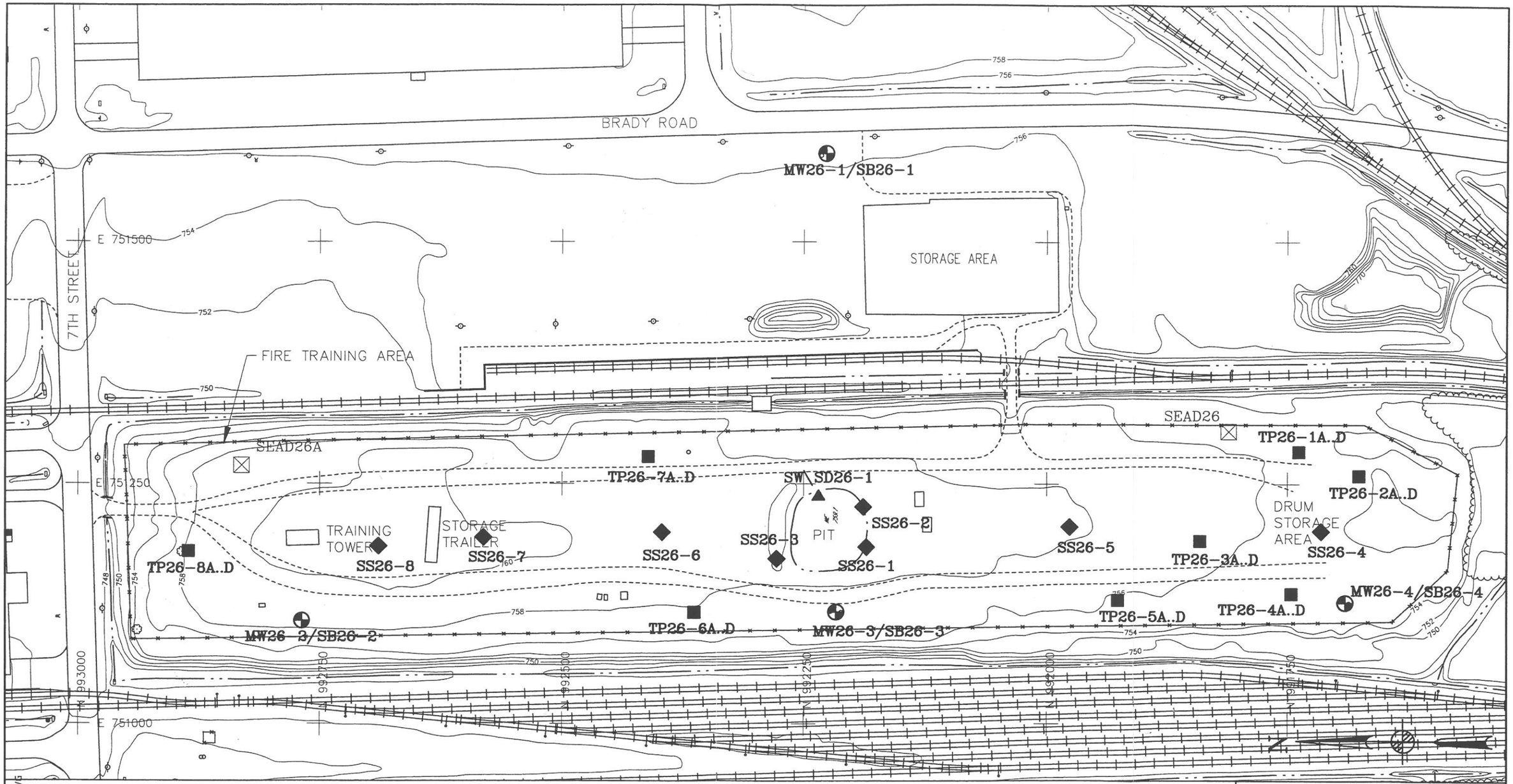
Comment 2: The last sentence in Section 5.2, Recommendations, indicates that the monitoring wells will be sampled during the 2021 five year review and decommissioned if trends remain the same. However, from the information presented in the Annual Report, the monitoring wells should not be decommissioned since exceedances of the New York State (NYS) Ambient Water Quality Criteria (AWQS) Class GA criteria (GA standard) and SEAD background concentrations are still present at the site. The monitoring wells should not be decommissioned until monitoring results show all analytes are below GA standards and EPA has approved discontinued monitoring. Further, it is unclear why samples are not proposed to be collected during the next five year review (i.e., 2016). Please revise the Annual Report to remove the statement regarding decommissioning of the monitoring wells. Please also indicate that the monitoring wells will be sampled during the next five year review or provide justification for why they are not proposed to be collected.

Response 2: The text was modified to match the recommendation to end sampling as proposed in the 2016 5-year review:

> Based on the current area-wide LUC prohibiting the use of groundwater within the PID Area (includes SEADs 16/17), the Army recommends concluding LTM at these sites because there is no planned future use of the groundwater. The wells will not be decommissioned at this time and sampling at these sites may take place in the future if the need arises (e.g., emerging contaminants, decisions during the 2021 5 Year Review). Annual LUC inspections will continue to insure that the groundwater is not accessed.

Comment 3: The discussion of trends in metals concentrations in Section 3.1.6, LTM Groundwater Data Trends, focuses on total results, but concentrations of iron and manganese measured in monitoring well MW16-5 during Year 7 exceeded groundwater standards in dissolved results and not total results. Since large differences between dissolved and total results have not been identified in recent years at SEAD-16, both dissolved and total results are considered representative of groundwater conditions. Therefore, these exceedances and trends in the iron and manganese dissolved results should be discussed. Please revise the Annual Report to discuss the trends in the total and dissolved concentrations that exceeded the groundwater standard for iron and manganese at SEAD-16.

Response 3: During Year 7, the iron (dissolved) concentration at well MW16-5 was uncharacteristically higher than the total result (360 µg/L and 280 µg/L, respectively). The dissolved and total results were approximately equivalent to the screening level; however, the dissolved value exceeded its screening level (300 µg/L).



ACAD\SENECA\7SWMU\ACADFIG\SD26SP.DWG

LEGEND

- MINOR WATERWAY
- MAJOR WATERWAY
- FENCE
- UNPAVED ROAD
- BRUSH LINE
- LANDFILL EXTENTS
- RAILROAD
- GROUND SURFACE ELEVATION CONTOUR (760)
- SURVEY MONUMENT
- ROAD SIGN
- DECIDOUS TREE
- FIRE HYDRANT
- MANHOLE
- GUIDE POST
- POLE
- UTILITY BOX
- CORDINATE GRID (250' GRID)
- OVERHEAD UTILITY POLE
- MAILBOX/RR SIGNAL
- MONITORING WELL
- SOIL BORING
- SEDIMENT SAMPLE
- SURFACE SOIL SAMPLE
- SURFACE WATER/SEDIMENT SAMPLE
- TEST PIT

50 0 50 100
(feet)

PARSONS
PARSONS ENGINEERING SCIENCE, INC.

CLIENT/PROJECT TITLE
SENECA ARMY DEPOT
EXPANDED SITE INSPECTION OF
7 HIGH-PRIORITY SWMU'S

DEPT. ENVIRONMENTAL ENGINEERING
Dwg. No. 720477-02000

FIGURE 2.8-2
SEAD-26 FIRE TRAINING PIT AND AREA
LOCATION OF SAMPLING POINTS

SCALE 1" = 100'
DATE MAY 1995
REV C

OVERBURDEN BORING REPORT

ENGINEERING–SCIENCE, INC.	CLIENT: ACOE	BORING NO.: MW26-2

PROJECT: 10 SWMU
LOCATION: SEAD 26

JOB NO.: 720477-01001
EST. GROUND ELEV.: 759.024
START DATE: 11/18/93
FINISH DATE: 11/18/93
CONTRACTOR: Empire
DRILLER: John
INSPECTOR: ES/LB
CHECKED BY:
CHECK DATE:

DRILLING SUMMARY:

DRILLING METHOD	HOLE DIA.	DEPTH INT.	SAMPLER SIZE	SAMPLER TYPE	HAMMER TYPE	HAMMER WT/FALL
HSA	8½"		3"x 2'	SS	HMR	140/30"

DRILLING ACRONYMS:

HSA	HOLLOW-STEM AUGERS	HMR	HAMMER	SS	SPLIT SPOON
DW	DRIVE-AND-WASH	SHR	SAFETY HAMMER	CS	CONTINUOUS SAMPLING
MRSLC	MUD-ROTARY SOIL-CORING	HHR	HYDRAULIC HAMMER	5I	5 FT INTERVAL SAMPLING
CA	CASING ADVANCER	DHR	DOWN-HOLE HAMMER	NS	NO SAMPLING
SPC	SPIN CASING	WL	WIRE-LINE	ST	SHELBY TUBE
				3S	3 INCH SPLIT SPOON

MONITORING EQUPMENT SUMMARY

INSTRUMENT TYPE	DETECTOR TYPE/ENERGY	RANGE	BACKGROUND READING	BACKGROUND TIME	BACKGROUND DATE	CALIBRATION TIME	CALIBRATION DATE	WEATHER
OVM		0-2000	0	0930	11/18/93			sunny.
Dust			.33	0930	11/18/93			

MONITORING ACRONYMS

PID	PHOTO - IONIZATION DETECTOR	BGD	BACKGROUND	DGRT	DRAEGER TUBES
FID	FLAME - IONIZATION DETECTOR	CPM	COUNTS PER MINUTE	PPB	PARTS PER BILLION
GMD	GEIGER MUELLER DETECTOR	PPM	PARTS PER MILLION	MDL	METHOD DETECTION LIMIT
SCT	SCINTILLATION DETECTOR	RAD	RADIATION		

COMMENTS:

OTHER REPORTS	DATE/PENDING	N/A
WELL DEVELOPMENT		
SURVEYOR		
CORE LOG		
WELL INSTALLATION DETAILS		
HYDRAULIC TESTING		
GEOPHYSICAL LOGGING		

SEE MASTER ACRONYM LIST FOR COMPLETE LISTING OF ABBREVIATIONS

BORING NO.:

OVERBURDEN BORING REPORT

ENGINEERING–SCIENCE, INC.	CLIENT: ACOE	BORING #: MW 26-2

MONITORING			
INSTRUMENT	INTERVAL	BGD	TIME
OVM	0-2000	0	0930
DUST	0-0.99	.33	0930

COMMENTS:

DRILLER: Empire/Kbh

INSPECTOR:

DATE:

DEPTH (FT)	SAMPLING: BLOWS PER 6 INCHES	SAMPLING: PENE-TRATION RANGE (FEET)	SAMPLING: RECOV-ERY RANGE (FEET)	SAMPLE: DEPTH INT (FEET)	SAMPLE: NO.	SAMPLE: VOC	SAMPLE: RAD SCRN	SAMPLE DESCRIPTION (As per Burmeister: color, grain size, MAJOR COMPONENT, Minor Components with amount modifiers and grain-size, density, stratification, wetness, etc.)	USCS CLASS	STRATUM CLASS
	8	0			26-			Topsoil		
1	12		1.7		2.1, 2.5, 2.1 MRD	0	X	gray weath. SHALE fill.		Fill
2	15									
	17	2								
	16	2			26-					
3	23		1.3		2.2	0	X	Lt. brown SILT, some Clay, some shale fragments (to 1.5" dia.) moist.		Fill
4	43									
	21	4								
	10	4			26-					
5	15		1.5		23	0	X	Lt. brown SILT, some CLAY, some shale fragments, dense.		
	14									
6	15	6								
	3	6			26-					
7	4		1.0		24	0	X	Lt brown SILT, some Clay, little Cobbles (to 2" dia - rounded) oxidation, trace fine sand, moist		
	6									
8	9	8								
	8	8			26-					
9	9		1.9		2.6	X	X			
	10									
10	15	10				*				
	6	10			26-					
11	7		2.0		2.7	X	X	Lt. brown fine SAND, some silt, oxidation, moist ← sand lense - med. sand		
	7									
12	9	12						Lt. brown SILT, some clay, little shale fragmts, oxidation, wet.		
	60/.4							weathered shale		
13										
								Augered to 14.0'		
14										
15										
20								* OVM not working		

SEE MASTER ACRONYM LIST FOR COMPLETE LISTING OF ABBREVIATIONS

BORING #:

OVERBURDEN BORING REPORT

ENGINEERING–SCIENCE, INC. | CLIENT: ACOE | BORING NO.: MW26-3

PROJECT: 10 SWMU

LOCATION: SEAD 26

JOB NO.: 20477-01001

EST. GROUND ELEV.: 751.567

START DATE: 11/8/93

FINISH DATE: 11/19/93

CONTRACTOR: Empire

DRILLER: John W.

INSPECTOR: ES

CHECKED BY:

CHECK DATE:

DRILLING SUMMARY:

DRILLING METHOD	HOLE DIA.	DEPTH INT.	SAMPLER SIZE	SAMPLER TYPE	HAMMER TYPE	HAMMER WT/FALL
HSA	8½"		2'x3"	SS	HMR	140#/30"

DRILLING ACRONYMS:

HSA	HOLLOW–STEM AUGERS	HMR	HAMMER	SS	SPLIT SPOON
DW	DRIVE–AND–WASH	SHR	SAFETY HAMMER	CS	CONTINUOUS SAMPLING
MRSLC	MUD–ROTARY SOIL–CORING	HHR	HYDRAULIC HAMMER	5I	5 FT INTERVAL SAMPLING
CA	CASING ADVANCER	DHR	DOWN–HOLE HAMMER	NS	NO SAMPLING
SPC	SPIN CASING	WL	WIRE–LINE	ST	SHELBY TUBE
				3S	3 INCH SPLIT SPOON

MONITORING EQUPMENT SUMMARY

INSTRUMENT TYPE	DETECTOR TYPE/ENERGY	RANGE	BACKGROUND READING	BACKGROUND TIME	BACKGROUND DATE	CALIBRATION TIME	CALIBRATION DATE	WEATHER
OVM			0.0	1450	11/18/93			Sunny.
Dust			0.04	1450	11/18/93			
OVM			0-0.1	800	11/19/93			cloudy
Dust			0	800	11/19/93			windy

MONITORING ACRONYMS

PID	PHOTO – IONIZATION DETECTOR	BGD	BACKGROUND	DGRT	DRAEGER TUBES
FID	FLAME – IONIZATION DETECTOR	CPM	COUNTS PER MINUTE	PPB	PARTS PER BILLION
GMD	GEIGER MUELLER DETECTOR	PPM	PARTS PER MILLION	MDL	METHOD DETECTION LIMIT
SCT	SCINTILLATION DETECTOR	RAD	RADIATION		

COMMENTS:

OTHER REPORTS	DATE/PENDING	N/A
WELL DEVELOPMENT		
SURVEYOR		
CORE LOG		
WELL INSTALLATION DETAILS		
HYDRAULIC TESTING		
GEOPHYSICAL LOGGING		

SEE MASTER ACRONYM LIST FOR COMPLETE LISTING OF ABBREVIATIONS

BORING NO.:

OVERBURDEN BORING REPORT

ENGINEERING–SCIENCE, INC. | CLIENT: ACOE | BORING #: MW26-3

MONITORING			
INSTRUMENT	INTERVAL	BGD	TIME
OVM		0.0	1450
DUST		.02	1450

COMMENTS:

DRILLER: Empire

INSPECTOR:

DATE: 11/18/93

DEPTH (FT)	SAMPLING BLOWS PER 6 INCHES	SAMPLING PENETRATION RANGE (FEET)	SAMPLING RECOVERY RANGE (FEET)	SAMPLE DEPTH INT (FEET)	SAMPLE NO.	SAMPLE VOC	SAMPLE RAD SCRN	SAMPLE DESCRIPTION (As per Burmeister: color, grain size, MAJOR COMPONENT, Minor Components with amount modifiers and grain–size, density, stratification, wetness, etc.)	USCS CLASS	STRATUM CLASS
								Topsoil		
1	6	0			26					
	9		1.7					Lt. brown SILT, some Clay, little Shale		
	12				3.1	0	X	fragments, moist, oxidation		
2	4	2								
	9	2								
3	9		1.6		26					
	7				3.2	0	X			
4	8	4								
	5	4								
5	5				26					
	7		1.6		3.3	0	X			
6	9	6								
	10	6			26					
7	10							Drk brown SILT, some Clay,		
	15		2.0		3.4	0.6	X	little Shale fragments, extensive oxidation,		
8	10	8						moist to wet		
	7	8								
9	9				26			AA, oxidation		
	10		1.5		3.5	0	X			
10	10	10								
	6	10			26	✓		AA, moist to wet		
11	10									
	29		1.7		36	0	X			
12	19	12						Gray weathered Shale, dry.		
	52									
13	100/.4							AA, wet.		
14								Spoon refusal @ 12.5'		
								Augered to 14.0'		
15										
20										

OVERBURDEN BORING REPORT

ENGINEERING–SCIENCE, INC. | CLIENT: ACOE | BORING NO.: MW26-4

PROJECT: 10 SWMU

LOCATION: SEAD 26

JOB NO.: 720477-01001

EST. GROUND ELEV.: 750.012

START DATE: 11/19/93

FINISH DATE: 11/19/93

CONTRACTOR: Empire

DRILLER: John

INSPECTOR: ES

CHECKED BY:

CHECK DATE:

DRILLING SUMMARY:

DRILLING METHOD	HOLE DIA.	DEPTH INT.	SAMPLER SIZE	SAMPLER TYPE	HAMMER TYPE	HAMMER WT/FALL
HSA	8½"		3" x 2'	SS	HMR	140#/30"

DRILLING ACRONYMS:

HSA	HOLLOW-STEM AUGERS	HMR	HAMMER	SS	SPLIT SPOON
DW	DRIVE-AND-WASH	SHR	SAFETY HAMMER	CS	CONTINUOUS SAMPLING
MRSLC	MUD-ROTARY SOIL-CORING	HHR	HYDRAULIC HAMMER	5I	5 FT INTERVAL SAMPLING
CA	CASING ADVANCER	DHR	DOWN-HOLE HAMMER	NS	NO SAMPLING
SPC	SPIN CASING	WL	WIRE-LINE	ST	SHELBY TUBE
				3S	3 INCH SPLIT SPOON

MONITORING EQUPMENT SUMMARY

INSTRUMENT TYPE	DETECTOR TYPE/ENERGY	RANGE	BACKGROUND READING	BACKGROUND TIME	BACKGROUND DATE	CALIBRATION TIME	CALIBRATION DATE	WEATHER
OVM			0	930	11/19/93			cloudy
Dust			0.45	930	11/19/93			windy

MONITORING ACRONYMS

PID	PHOTO - IONIZATION DETECTOR	BGD	BACKGROUND	DGRT	DRAEGER TUBES
FID	FLAME - IONIZATION DETECTOR	CPM	COUNTS PER MINUTE	PPB	PARTS PER BILLION
GMD	GEIGER MUELLER DETECTOR	PPM	PARTS PER MILLION	MDL	METHOD DETECTION LIMIT
SCT	SCINTILLATION DETECTOR	RAD	RADIATION		

COMMENTS:

OTHER REPORTS	DATE/PENDING	N/A
WELL DEVELOPMENT		
SURVEYOR		
CORE LOG		
WELL INSTALLATION DETAILS		
HYDRAULIC TESTING		
GEOPHYSICAL LOGGING		

SEE MASTER ACRONYM LIST FOR COMPLETE LISTING OF ABBREVIATIONS

BORING NO.:

OVERBURDEN BORING REPORT

ENGINEERING–SCIENCE, INC.	CLIENT: ACOE	BORING #: MW26-4

MONITORING			
INSTRUMENT	INTERVAL	BGD	TIME
OVM		0.0	9.30
Dust		0.45	930

COMMENTS:

DRILLER: Empire
INSPECTOR: ES
DATE: 11/19/93

DEPTH (FT)	SAMPLING: BLOWS PER 6 INCHES	SAMPLING: PENE-TRATION RANGE (FEET)	SAMPLING: RECOV-ERY RANGE (FEET)	SAMPLE: DEPTH INT (FEET)	SAMPLE: NO.	SAMPLE: VOC	SAMPLE: RAD SCRN	SAMPLE DESCRIPTION (As per Burmeister: color, grain size, MAJOR COMPONENT, Minor Components with amount modifiers and grain-size, density, stratification, wetness, etc.)	USCS CLASS	STRATUM CLASS
1	7 12 12 9		1.2		26 4.1	0	X	Topsoil weath Shale fill		
3	9 8 9 6		1.4		26 4.2	0	X	Med brown SILT, and wash Shale fragments (to 2" dia.) moist.		Fill
5	8 9 10 12		1.4		26 4.3	0	X			
7	11 10 17 24		2.0		26 4.4	0		Med brown SILT, some clay, little Shale fragments (.25") moist		
9	38 100		1.0		26 4.5	0		weathered Shale, dry		
10								Spoon refusal @ 9.0' Augered To 11.5'		
15										
20										

SEE MASTER ACRONYM LIST FOR COMPLETE LISTING OF ABBREVIATIONS

BORING #:

SITE 122E

56(6)PR

SB122D-02

SB122D-01

AIRCRAFT PARKING AND FUELING

CLEAR

OVERRUN

MW122E-1/SB122E-1

AIRCRAFT FUELING (SOUTH)

500' LATERAL CLEARANCE

115Q-X

2307

SS122A-5

SS122A-4

SS122A-3

SS122A-2

SS122A-1

SURFACE SLOPE RATIO 7:1

2308

SS122B-3

SS122B-2

SS122B-5

SS122B-4

SS122B-1

2301

TRANSITIONAL

RANGE 1

RANGE 2

2302

2311

NORTH-WEST SOUTH-EAST RUNWAY

AIRCRAFT PARKING AND FUELING (CENTRAL)

2310

2305

2304

2303

MW122E-2/SB122E-2

2315

8(4)PS/PR

S-2306

7(2)PS

6(4)PS/PR

NEW YORK STATE HIGHWAY

AIRCRAFT FUELING (NORTH)

500' LATERAL CLEARANCE

MW122E-3/SB122E-3

2(1)

R:\SENECA\EBS\ST-122E.DWG

N

LEGEND:

SB123B-1 — SOIL BORING

SS123B-1 — SURFACE SOIL SAMPLE

MW122E-1 — TRMPORARY MONITORING WELL

BRAC PARCEL LABEL DEFINITIONS

8(2)PS

- PARCEL NUMBER
- CATEGORY NUMBER
- CONTAMINATION DESCRIPTION:
  - PS PETROLEUM STORAGE
  - PR PETROLEUM RELEASE OR DISPOSAL
  - HS HAZARDOUS SUBSTANCE STORAGE
  - HR HAZARDOUS SUBSTANCE RELEASE OR DISPOSAL
  - (P) POSSIBLE (UNVERIFIED)

NON-CERCLA ISSUE (QUALIFIED) LABEL DEFINITIONS

8-19Q-A(P)

- PARCEL NUMBER
- FACILITY NUMBER (IF APPLICABLE)
- QUALIFIED
- QUALIFIERS:
  - A ASBESTOS-CONTAINING MATERIAL
  - L LEAD-BASED PAINT
  - P PCB
  - R RADON
  - X UXO AND/OR ORDNANCE FRAGMENTS
  - RD RADIONUCLIDES
  - (P) POSSIBLE (UNVERIFIED)

-500 0 500

SCALE: 1" = 500'

PARSONS

PARSONS ENGINEERING SCIENCE, INC.

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT ACTIVITY

ENVIROMENTAL BASELINE SURVEY

INVESTIGATION OF NON-EVALUATED SITES

DEPT. ENVIRONMENTAL ENGINEERING | Dwg. No.

FIGURE 7-1

SITE FEATURES AND SAMPLE LOCATIONS AT EBS SITE 122E DEICING PLANES

SCALE 1" = 500' | DATE FEBRUARY 1999 | REV A

LOG OF BORING 122D-1

PROJECT: Seneca Non-evaluated EBS Sites
PROJECT LOCATION: Seneca Army Depot, Romulus, New York
ASSOCIATED AREA/UNIT: SEAD 122
PROJECT NO: 733193-01001
DATE STARTED: 3/05/98
DATE COMPLETED: 3/05/98
DRILLING CONTRACTOR: Nothnagle
DRILLING METHOD: HSA 8"
SAMPLING METHOD: Split Spoon

TOTAL DEPTH: 13.9
DEPTH TO WATER: 12.5
BORING LOCATION: 987911.494 ft NORTH
741222.1228 ft EAST
COORDINATE SYSTEM: NAD83
GROUND SURFACE ELEVATION: 644.8973 ft
ELEVATION DATUM: NAVD88
INSPECTOR: MW
CHECKED BY: ITR

Sample Number	Blow Counts (# Blows per 6")	Sample Recovery	VOC Screen-PID (ppm)	Depth (ft)	Macro Lithology	This log is part of a report prepared by Parsons Engineering-Science, Inc. for the named company and should be read together with the report for complete interpretation. This summary applies only at the location of this boring and at the time of drilling. Subsurface conditions may differ at other locations. DESCRIPTION	USCS
EB201	6 7 7 8	1.5	0	0		Light Brown, CLAY, some Silt, little +fine to coarse Gravel, moist. Roots in top 2"	CL
				1.5		No Recovery	
	5 9 15 20	1.3	0	2		Light Brown to Greenish Gray, CLAY, and -Silt, trace -fine Sand, little fine to coarse Gravel, moist.	CL
				3.3		No Recovery	
	9 15 25 27	1.8	0	4		Light Brown to Greenish Gray, SILT, some +Clay, little -fine Sand, little fine to coarse Gravel, moist.	ML
				5.8		No Recovery	
EB202	13 25 25 25	1.7	0	6		Light Brown, CLAY, some Silt, trace fine Sand, little +Gravel, moist.	CL
				6.8		Light Brown to Greenish Gray, SILT, little +fine Sand, some -fine to coarse Gravel, trace Clay, wet.	
				7.7		No Recovery	
	19 33 41 50	2	0	8		Light Brown, Silt, trace fine Sand, some fine to coarse Gravel, wet.	ML
				9			

NOTES:

UNITED STATES ARMY
CORPS OF ENGINEERS
Seneca Army Depot
Romulus, New York

LOG OF BORING 122D-1

Sheet 1 of 2

LOG OF BORING 122D-1

PROJECT: Seneca Non-evaluated EBS Sites
PROJECT LOCATION: Seneca Army Depot, Romulus, New York
ASSOCIATED AREA/UNIT: SEAD 122
PROJECT NO: 733193-01001
DATE STARTED: 3/05/98
DATE COMPLETED: 3/05/98
DRILLING CONTRACTOR: Nothnagle
DRILLING METHOD: HSA 8"
SAMPLING METHOD: Split Spoon

TOTAL DEPTH: 13.9
DEPTH TO WATER: 12.5
BORING LOCATION: 987911.494 ft NORTH
741222.1228 ft EAST
COORDINATE SYSTEM: NAD83
GROUND SURFACE ELEVATION: 644.8973 ft
ELEVATION DATUM: NAVD88
INSPECTOR: MW
CHECKED BY: ITR

Sample Number	Blow Counts (# Blows per 6")	Sample Recovery	VOC Screen-PID (ppm)	Depth (ft)	Macro Lithology	This log is part of a report prepared by Parsons Engineering-Science, Inc. for the named company and should be read together with the report for complete interpretation. This summary applies only at the location of this boring and at the time of drilling. Subsurface conditions may differ at other locations.	USCS
						DESCRIPTION	
	37 85 100/.2	1.2	0	10 10 11 11.2		Light Brown, SILT, trace fine Sand, some +fine to coarse Gravel, moist to wet.	ML
				12		No Recovery	
	27 55 90 100/.1	1.5	0	12 13 13.5		Olive Gray, SILT, trace -fine Sand, some +fine to coarse Gravel, saturated.	TL
				13.9		Wheathered SHALE.	
						Auger Refusal at 13.9'.	

NOTES:

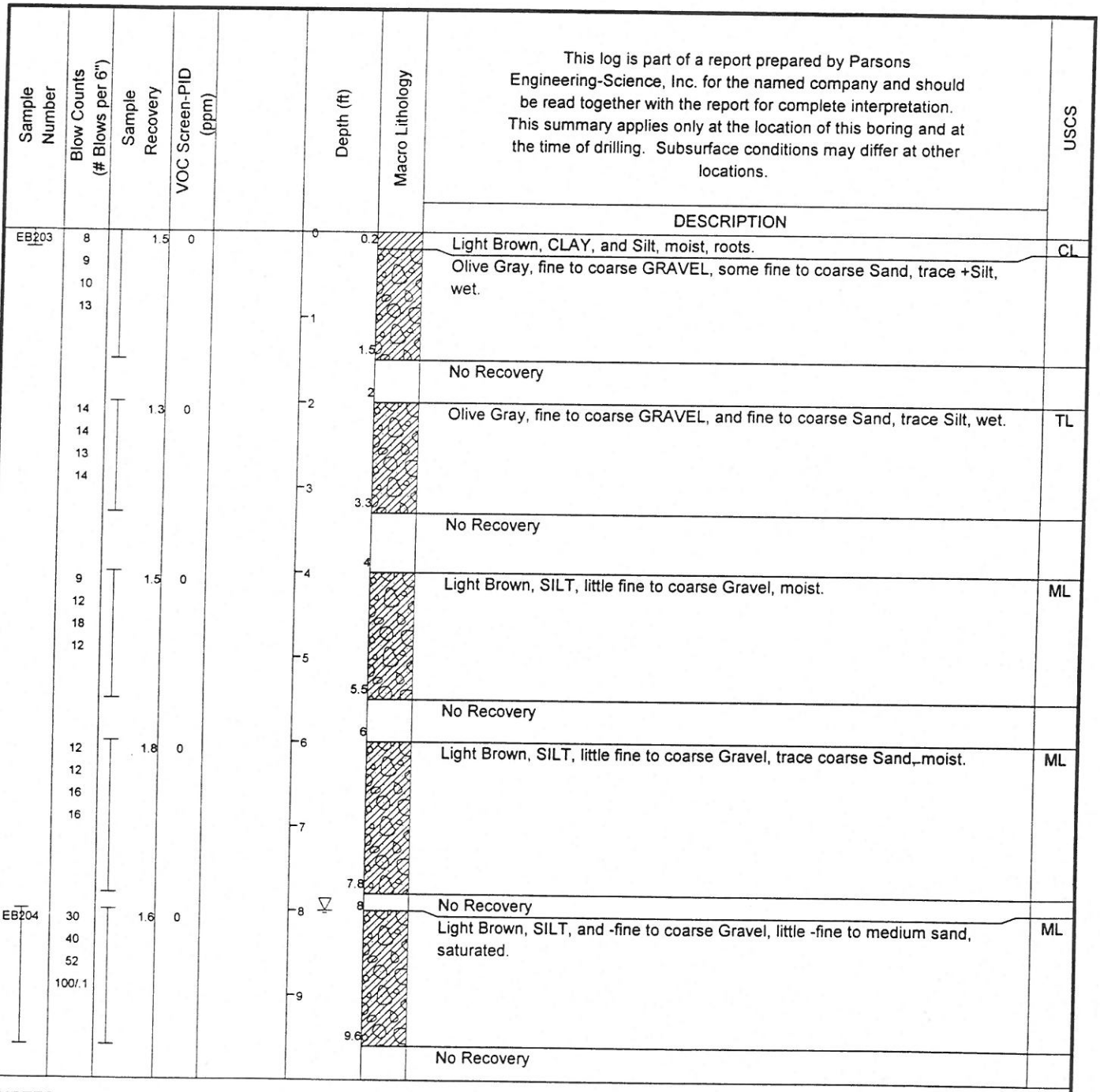
UNITED STATES ARMY
CORPS OF ENGINEERS
Seneca Army Depot
Romulus, New York

LOG OF BORING 122D-1

LOG OF BORING 122D-2

PROJECT: Seneca Non-evaluated EBS Sites
PROJECT LOCATION: Seneca Army Depot, Romulus, New York
ASSOCIATED AREA/UNIT: SEAD 122
PROJECT NO: 733193-01001
DATE STARTED: 3/05/98
DATE COMPLETED: 3/05/98
DRILLING CONTRACTOR: Nothnagle
DRILLING METHOD: HSA 8"
SAMPLING METHOD: Split Spoon

TOTAL DEPTH: 14
DEPTH TO WATER: 8
BORING LOCATION: 987799.2085 ft NORTH
741278.0134 ft EAST
COORDINATE SYSTEM: NAD83
GROUND SURFACE ELEVATION: 643.8361 ft
ELEVATION DATUM: NAVD88
INSPECTOR: MW
CHECKED BY: ITR



NOTES:

UNITED STATES ARMY
CORPS OF ENGINEERS
Seneca Army Depot
Romulus, New York

LOG OF BORING 122D-2

LOG OF BORING 122D-2

PROJECT: Seneca Non-evaluated EBS Sites
PROJECT LOCATION: Seneca Army Depot, Romulus, New York
ASSOCIATED AREA/UNIT: SEAD 122
PROJECT NO: 733193-01001
DATE STARTED: 3/05/98
DATE COMPLETED: 3/05/98
DRILLING CONTRACTOR: Nothnagle
DRILLING METHOD: HSA 8"
SAMPLING METHOD: Split Spoon

TOTAL DEPTH: 14
DEPTH TO WATER: 8
BORING LOCATION: 987799.2085 ft NORTH
741278.0134 ft EAST
COORDINATE SYSTEM: NAD83
GROUND SURFACE ELEVATION: 643.8361 ft
ELEVATION DATUM: NAVD88
INSPECTOR: MW
CHECKED BY: ITR

Sample Number	Blow Counts (# Blows per 6")	Sample Recovery	VOC Screen-PID (ppm)	Depth (ft)	Macro Lithology	This log is part of a report prepared by Parsons Engineering-Science, Inc. for the named company and should be read together with the report for complete interpretation. This summary applies only at the location of this boring and at the time of drilling. Subsurface conditions may differ at other locations.	USCS
						DESCRIPTION	
	28 46 80 100/.5	1.8	0.2	10 11		Light Brown, SILT, and -fine to coarse Gravel, little -fine to coarse Sand, saturated.	ML
				11.8		No Recovery	
	29 43 100/.3	1.3	0.2	12 13		Olive Gray, fine to coarse GRAVEL, some Silt, trace +fine to coarse Sand, saturated.	GM
				13.3		Weathered SHALE.	
				14		Auger Refusal at 14.0'.	

NOTES:

UNITED STATES ARMY
CORPS OF ENGINEERS
Seneca Army Depot
Romulus, New York

LOG OF BORING 122D-2

Sheet 2 of 2

LOG OF BORING 122E-1

PROJECT: Seneca Non-evaluated EBS Sites
PROJECT LOCATION: Seneca Army Depot, Romulus, New York
ASSOCIATED AREA/UNIT: SEAD 122
PROJECT NO: 733193-01001
DATE STARTED: 3/06/98
DATE COMPLETED: 3/06/98
DRILLING CONTRACTOR: Nothnagle
DRILLING METHOD: HSA 8"
SAMPLING METHOD: Split Spoon

TOTAL DEPTH: 9.1
DEPTH TO WATER: 7.2
BORING LOCATION: 987033.7607 ft NORTH
740754.7201 ft EAST
COORDINATE SYSTEM: NAD83
GROUND SURFACE ELEVATION: 638.9787 ft
ELEVATION DATUM: NAVD88
INSPECTOR: DRG
CHECKED BY: ITR

Sample Number	Blow Counts (# Blows per 6")	Sample Recovery	VOC Screen-PID (ppm)	Depth (ft)	Macro Lithology	DESCRIPTION	USCS
						This log is part of a report prepared by Parsons Engineering-Science, Inc. for the named company and should be read together with the report for complete interpretation. This summary applies only at the location of this boring and at the time of drilling. Subsurface conditions may differ at other locations.	
EB205	4 14 8 14	1.5	0	0 1 1.5		Olive Gray, SILT, little coarse Sand, trace fine Gravel, moist.	OL
				2		No Recovery	
	10 12 15 30	1.8	0	2 3 3.8		Olive Gray, SILT, some Clay, little fine Sand, trace Cobble, wet.	CL
				4		No Recovery	
	20 100/.1	0.6	0	4 4.6		Olive Gray, fine SAND, some medium Gravel, little Cobble, trace Silt, moist.	SP
				5 6		No Recovery	
EB207	22 87 100/.5	1.5	0	6 7 7.5		Brown fine to medium, SAND, some finer to coarse Gravel, some Cobble, trace Silt, saturated.	TL
				8		No Recovery.	
	100/.5	0.5	0	8 8.2 8.5		Olive Gray, SILT, Shale fragments. Competant Shale.	TL
				9		No Recovery.	
				9		Auger Refusal at 9.0'.	

NOTES:

UNITED STATES ARMY
CORPS OF ENGINEERS
Seneca Army Depot
Romulus, New York

LOG OF BORING 122E-1

Sheet 1 of 1

LOG OF BORING 122E-2

PROJECT: Seneca Non-evaluated EBS Sites
PROJECT LOCATION: Seneca Army Depot, Romulus, New York
ASSOCIATED AREA/UNIT: SEAD 122
PROJECT NO: 733193-01001
DATE STARTED: 3/06/98
DATE COMPLETED: 3/06/98
DRILLING CONTRACTOR: Nothnagle
DRILLING METHOD: HSA 8"
SAMPLING METHOD: Split Spoon

TOTAL DEPTH: 12.5
DEPTH TO WATER: 2.2
BORING LOCATION: 988958.412 ft NORTH
739018.1027 ft EAST
COORDINATE SYSTEM: NAD83
GROUND SURFACE ELEVATION: 602.0001 ft
ELEVATION DATUM: NAVD88
INSPECTOR: DRG
CHECKED BY: ITR

Sample Number	Blow Counts (# Blows per 6")	Sample Recovery	VOC Screen-PID (ppm)	Depth (ft)	Macro Lithology	DESCRIPTION	USCS
						This log is part of a report prepared by Parsons Engineering-Science, Inc. for the named company and should be read together with the report for complete interpretation. This summary applies only at the location of this boring and at the time of drilling. Subsurface conditions may differ at other locations.	
EB208	7 11 11 14	1.5	0	0		Brown, SILT, trace fine Sand, little organics, trace coarse Gravel, trace Cobble, moist.	FL
				1			
				1.5		Olive Gray, fine SAND, little coarse Sand to fine Gravel, trace Cobble, trace Silt, iron oxide viens, moist.	
						No Recovery.	
				2			
EB209	13 13 21 13	0.8	0	2 ▽		Brown, coarse SAND and fine GRAVEL, little fine to medium Sand, trace Cobbles, wet to saturated.	FL
				2.8		No Recovery	
				3			
				4			
	7 7 8 22	1.5	0	4		Brown, coarse SAND and fine GRAVEL, little fine to medium Sand, trace Cobbles, wet to saturated.	FL
				5			
				5.5		Olive Gray, SILT and very fine SAND, little coarse Sand to fine Gravel, trace Cobble, iron oxide veins, saturated.	
						No Recovery.	
				6			
	32 100/.5	0.5	0	6		Olive Gray, SILT and very fine SAND, little coarse Sand to fine Gravel, trace Cobble, iron oxide veins, saturated.	TL
				6.5		No Recovery.	
				7			
				8			
	100/.5	0.3	0	8		Olive Gray, SHALE chips, some Silt and fine Sand, weathered Shale, wet.	BRK
				8.3		No Recovery.	
				9			

NOTES:

UNITED STATES ARMY
CORPS OF ENGINEERS
Seneca Army Depot
Romulus, New York

LOG OF BORING 122E-2

Sheet 1 of 2

LOG OF BORING 122E-2

PROJECT: Seneca Non-evaluated EBS Sites
PROJECT LOCATION: Seneca Army Depot, Romulus, New York
ASSOCIATED AREA/UNIT: SEAD 122
PROJECT NO: 733193-01001
DATE STARTED: 3/06/98
DATE COMPLETED: 3/06/98
DRILLING CONTRACTOR: Nothnagle
DRILLING METHOD: HSA 8"
SAMPLING METHOD: Split Spoon

TOTAL DEPTH: 12.5
DEPTH TO WATER: 2.2
BORING LOCATION: 988958.412 ft NORTH
739018.1027 ft EAST
COORDINATE SYSTEM: NAD83
GROUND SURFACE ELEVATION: 602.0001 ft
ELEVATION DATUM: NAVD88
INSPECTOR: DRG
CHECKED BY: ITR

Sample Number	Blow Counts (# Blows per 6")	Sample Recovery	VOC Screen-PID (ppm)	Depth (ft)	Macro Lithology	This log is part of a report prepared by Parsons Engineering-Science, Inc. for the named company and should be read together with the report for complete interpretation. This summary applies only at the location of this boring and at the time of drilling. Subsurface conditions may differ at other locations.	USCS
						DESCRIPTION	
	12	0.4	0	10		Olive Gray, SHALE chips, some Silt and fine Sand, weathered Shale, wet.	BRK
	22			10.4		No Recovery.	
	66						
	100/.4			11			
				12			
	100/.5		0			No Recovery	
				12.5		Auger refusal at 12.5'.	

NOTES:

UNITED STATES ARMY
CORPS OF ENGINEERS
Seneca Army Depot
Romulus, New York

LOG OF BORING 122E-2

Sheet 2 of 2

LOG OF BORING 122E-3

PROJECT: Seneca Non-evaluated EBS Sites
PROJECT LOCATION: Seneca Army Depot, Romulus, New York
ASSOCIATED AREA/UNIT: SEAD 122
PROJECT NO: 733193-01001
DATE STARTED: 3/06/98
DATE COMPLETED: 3/06/98
DRILLING CONTRACTOR: Nothnagle
DRILLING METHOD: HSA 8"
SAMPLING METHOD: Split Spoon

TOTAL DEPTH: 11.8
DEPTH TO WATER: 2.4
BORING LOCATION: 991432.0738 ft NORTH
738522.1617 ft EAST
COORDINATE SYSTEM: NAD83
GROUND SURFACE ELEVATION: 609.7340 ft
ELEVATION DATUM: NAVD88
INSPECTOR: DRG
CHECKED BY: ITR

Sample Number	Blow Counts (# Blows per 6")	Sample Recovery	VOC Screen-PID (ppm)	Depth (ft)	Macro Lithology	This log is part of a report prepared by Parsons Engineering-Science, Inc. for the named company and should be read together with the report for complete interpretation. This summary applies only at the location of this boring and at the time of drilling. Subsurface conditions may differ at other locations.	USCS
						DESCRIPTION	
EB210	20 18 7 7	1.2	0	0 1 1.2		Dark Brown to reddish Brown, SILT, Some fine to medium sand, littlefine Gravel, trace Clay and Cobbles, moist.	ML
						No Recovery.	
EB211	15 13 8 8	0.5	0	2 2.5		Light Brown, fine GRAVEL and Coarse SAND, little fine Sand, little coarse Gravel, little Cobble, wet.	GP
				3		No Recovery.	
	16 18 12 100/.2	1.5	0	4 4.5 5 5.5		Light Brown, fine GRAVEL and coarse SAND, little fine to medium Sand,, little coarse Gravel, little Cobble, wet.	GP
						Olive Gray to Brown, SILT, little coarse Sand to medium Gravel, trace cobbles, very tight till, iron oxide nodes.	
				6		No Recovery.	
	18 43 22 11	1	0	7		Olive Gray to Brown, SILT, little coarse Sand to medium Gravel, trace Cobbles, very tight till, iron oxide nodes.	TL
				8		No Recovery.	
	100/.4	0.4	0	8.4		Weathered SHALE.	BRK
				9		No Recovery.	

NOTES:

UNITED STATES ARMY
CORPS OF ENGINEERS
Seneca Army Depot
Romulus, New York

LOG OF BORING 122E-3

Sheet 1 of 2

LOG OF BORING 122E-3

PROJECT: Seneca Non-evaluated EBS Sites
PROJECT LOCATION: Seneca Army Depot, Romulus, New York
ASSOCIATED AREA/UNIT: SEAD 122
PROJECT NO: 733193-01001
DATE STARTED: 3/06/98
DATE COMPLETED: 3/06/98
DRILLING CONTRACTOR: Nothnagle
DRILLING METHOD: HSA 8"
SAMPLING METHOD: Split Spoon

TOTAL DEPTH: 11.8
DEPTH TO WATER: 2.4
BORING LOCATION: 991432.0738 ft NORTH
738522.1617 ft EAST
COORDINATE SYSTEM: NAD83
GROUND SURFACE ELEVATION: 609.7340 ft
ELEVATION DATUM: NAVD88
INSPECTOR: DRG
CHECKED BY: ITR

Sample Number	Blow Counts (# Blows per 6")	Sample Recovery	VOC Screen-PID (ppm)	Depth (ft)	Macro Lithology	This log is part of a report prepared by Parsons Engineering-Science, Inc. for the named company and should be read together with the report for complete interpretation. This summary applies only at the location of this boring and at the time of drilling. Subsurface conditions may differ at other locations.	USCS
						DESCRIPTION	
	100/.3	0.3	0	10		Compentant SHALE.	BRK
				10.3		No Recovery.	
				11			
				11.8		Auger Refusal at 11.8'.	

NOTES:

UNITED STATES ARMY
CORPS OF ENGINEERS
Seneca Army Depot
Romulus, New York

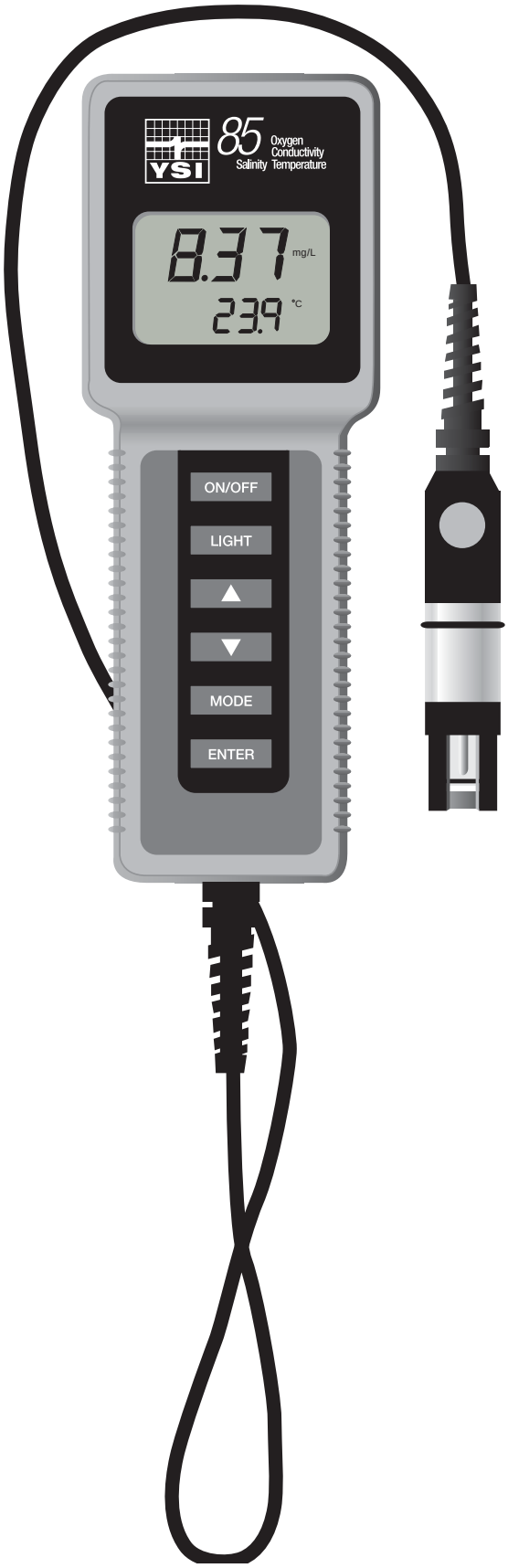
LOG OF BORING 122E-3

Appendix F

Equipment Manuals

Equipment Manuals are provided on the electronic (CD) version of this report.

YSI *incorporated*



YSI Model 85

Handheld Oxygen, Conductivity, Salinity, and Temperature System

Operations Manual

CONTENTS

SECTION 1 INTRODUCTION

The YSI Model 85 Handheld Dissolved Oxygen, Conductivity, Salinity and Temperature System is a rugged, micro-processor based, digital meter with an attached YSI combination conductivity and dissolved oxygen probe.

The YSI Model 85 is designed for use in field, lab, and process control applications as well as for environmental, aquaculture, and industrial uses. The Model 85 is available with cable lengths of either 10, 25, 50 or 100 feet. The body of the probe has been manufactured with stainless steel to add rugged durability and sinking weight. The probe also utilizes our easy to install cap membranes for measuring dissolved oxygen.

The YSI Model 85 probe is a non-detachable, combination sensor designed specifically for the YSI Model 85 Handheld System. The conductivity portion is a four-electrode cell with a cell constant of 5.0/cm ±4%. The dissolved oxygen portion is a polargraphic Clark type sensor.

The Model 85's microprocessor allows the system to be easily calibrated for dissolved oxygen or conductivity with the press of a few buttons. Additionally, the microprocessor performs a self-diagnostic routine each time the instrument is turned on. The self-diagnostic routine provides you with useful information about the conductivity cell constant and function of the instrument circuitry. The system simultaneously displays temperature (in °C), along with one of the following parameters: dissolved oxygen in either mg/L (milligrams per liter) or % air saturation; conductivity; temperature compensated conductivity; (in µS/cm or mS/cm), and salinity (in parts per thousand {ppt}).

The system requires only a single calibration regardless of which dissolved oxygen display you use. The calibration of conductivity is not required but is available. A single calibration will adjust the instrument, regardless if you are reading conductivity or temperature compensated conductivity. You can switch between all of these parameters with the push of a single key.

A calibration\storage chamber is built into the instrument case. A small sponge in the chamber can be moistened to provide a water saturated air environment that is ideal for air calibration of the dissolved oxygen probe. This chamber also provides a convenient place to store the probe when the system is not in use, and provides protection for the electrodes within the conductivity probe. The Model 85 case is also waterproof (rated to IP65). You can operate your Model 85 in the rain without damage to the instrument.

Six AA-size alkaline batteries power the instrument. A new set of alkaline batteries will provide approximately 100 hours of continuous operation. When batteries need to be replaced, the LCD will display a **"LO BAT"** message.

SECTION 2 PREPARING THE METER

2.1 UNPACKING

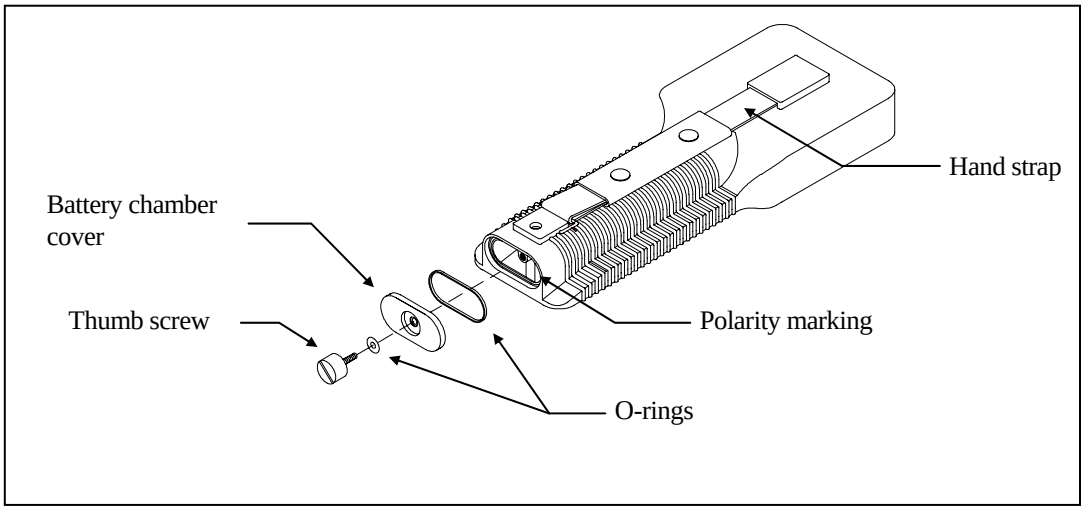
When you unpack your new YSI Model 85 Handheld Dissolved Oxygen, Conductivity, Salinity and Temperature System for the first time, check the packing list to make sure you have received everything you should have. If there is anything missing or damaged, call the dealer from whom you purchased the Model 85. If you do not know which of our authorized dealers sold the system to you, call YSI Customer Service at 800-765-4974 or 937-767-7241, and we'll be happy to help you.

2.2 WARRANTY CARD

Before you do anything else, please complete the Warranty Card and return it to YSI. This will record your purchase of this quality instrument in our computer system. Once your purchase is recorded, you will receive prompt, efficient service in the event any part of your YSI Model 85 should ever need repair and we will be able to quickly verify the warranty period.

2.3 BATTERIES

There are a few things you must do to prepare your YSI Model 85 for use. First, locate the six AA-size alkaline batteries that were included in your purchase. Use a screwdriver or a small coin to remove the thumbscrew on the bottom of the instrument. This thumbscrew holds the battery-chamber cover in place. The battery-chamber cover is marked with the words "OPEN" and "CLOSE."



NOTE: On some models, the battery cover thumbscrew may be unscrewed by hand (a screwdriver may not be required).

There is a small label inside each of the two battery-chamber sleeves. These labels illustrate the correct way to install the batteries into each sleeve of the battery-chamber.

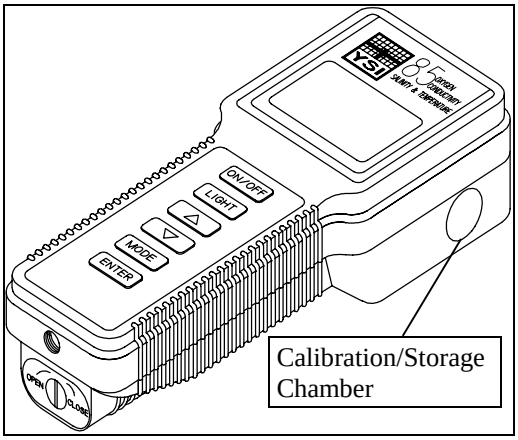
NOTE: It is very important that the batteries be installed ONLY as illustrated. The instrument will not function and may be damaged if the batteries are installed incorrectly.

Turn the instrument on by pressing and releasing the **ON/OFF** button on the front of the instrument. The liquid crystal display (LCD) should come on. Allow a few seconds for the instrument to complete its diagnostic routine. Notice that the instrument will display the specific cell constant of the conductivity probe during this diagnostic routine. If the instrument does not operate, consult the section entitled Troubleshooting.

You may also want to take the instrument into a dark room and with the instrument ON, hold down the **LIGHT** button. The instrument backlight should illuminate the LCD so that the display can be easily read.

2.4 CALIBRATION/STORAGE CHAMBER

The Model 85 has a convenient calibration storage chamber built into the instruments' side. This chamber provides an ideal storage area for the probe during transport and extended non-use. If you look into the chamber you should notice a small round sponge in the bottom of the chamber. Carefully put 3 to 6 drops of clean water into the sponge. Turn the instrument over and allow any excess water to drain out of the chamber. The wet sponge creates a 100% water saturated air environment for the probe, which is ideal for dissolved oxygen calibration.



2.5 HAND STRAP

The hand strap is designed to allow comfortable operation of the Model 85 with minimum effort. If the hand strap is adjusted correctly, it is unlikely that the instrument will be easily dropped or bumped from your hand. See figure on previous page.

To adjust the hand strap on the back of the meter, unsnap the vinyl cover and pull the two Velcro strips apart. Place your hand between the meter and the strap and adjust the strap length so that your hand is snugly held in place. Press the two Velcro strips back together and snap the vinyl cover back into place.

2.6 THE METER CASE

The meter case is sealed at the factory and is not intended to be opened, except by authorized service technicians. Do not attempt to separate the two halves of the meter case as this may damage the instrument, break the waterproof seal, and will void the manufacturer's warranty.

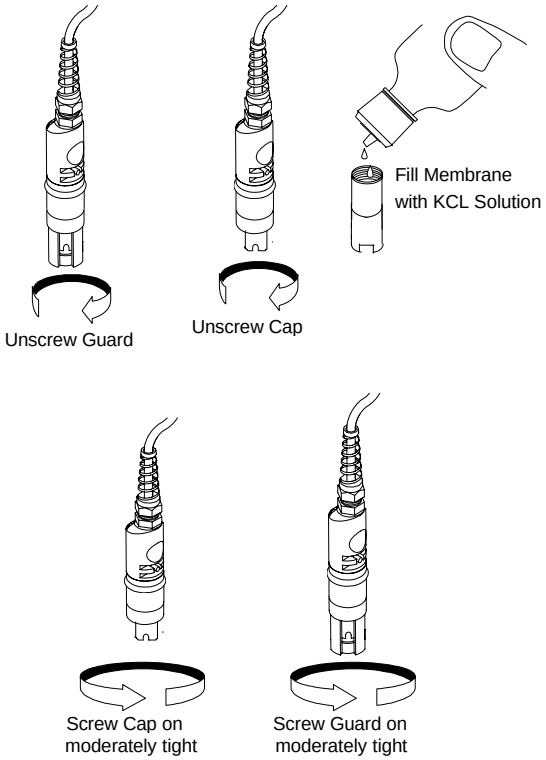
SECTION 3 PREPARING THE PROBE

The YSI Model 85 dissolved oxygen probe is shipped dry. The protective membrane cap on the probe tip must be removed and replaced with KCl solution and a new membrane cap before using the probe. Follow the instructions below to install KCl solution and the new membrane cap.

3.1 MEMBRANE CAP INSTALLATION

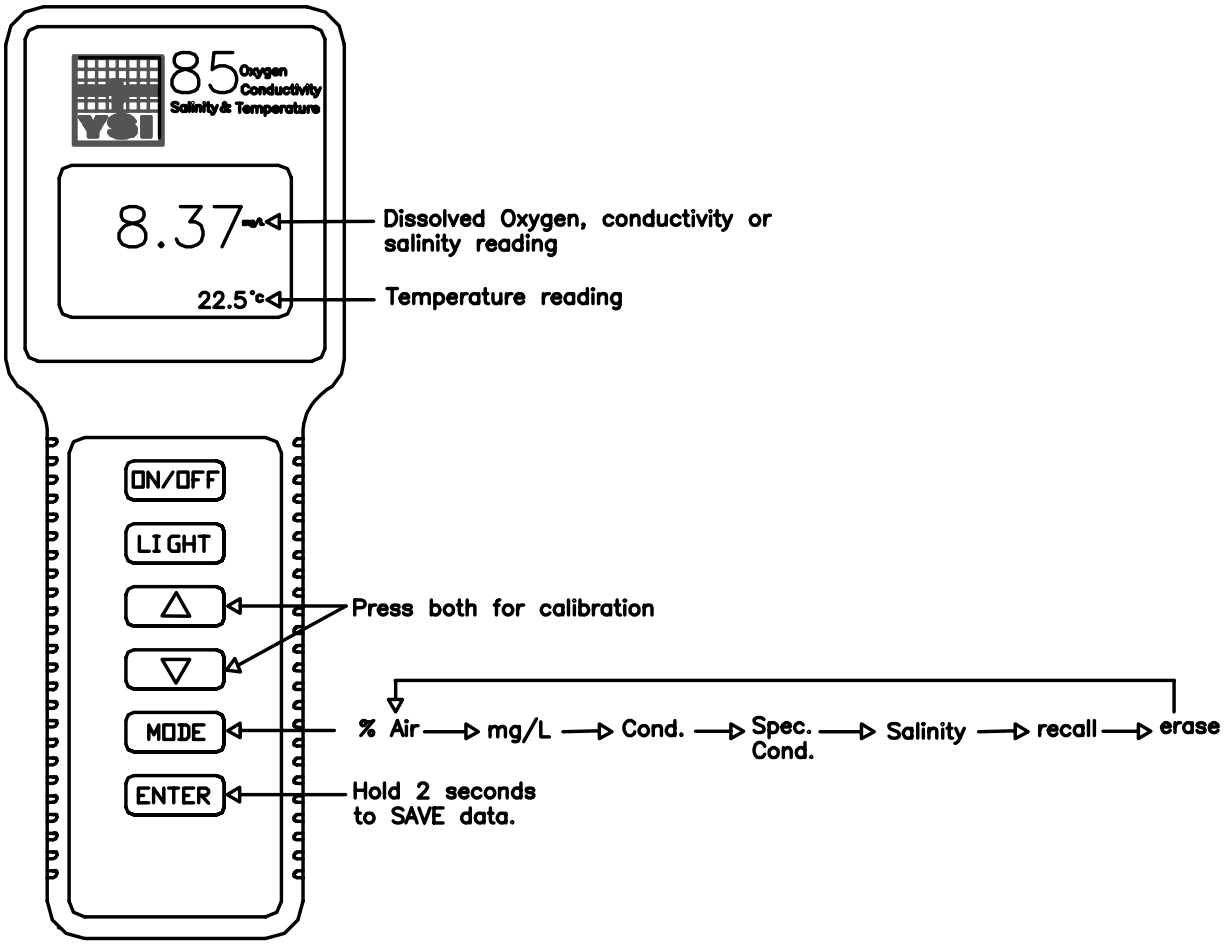
To install a new membrane on your YSI Model 85 dissolved oxygen probe:

1. Unscrew and remove the probe sensor guard.
2. Unscrew and remove the old membrane cap.
3. Thoroughly rinse the sensor tip with distilled water.
4. Prepare the electrolyte according to the directions on the KCl solution bottle.
5. Hold the membrane cap and fill it at least 1/2 full with the electrolyte solution.
6. Screw the membrane cap onto the probe moderately tight. A small amount of electrolyte should overflow.
7. Screw the probe sensor guard on moderately tight.



SECTION 4 OVERVIEW OF OPERATION

The following diagram is an overview of the operation of the Model 85. See the following sections for details of operation.

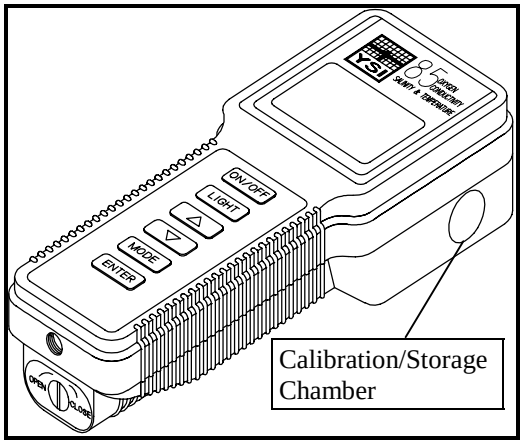


SECTION 5 CALIBRATION

5.1 CALIBRATION OF DISSOLVED OXYGEN

To accurately calibrate the YSI Model 85 you will need to know the approximate altitude of the region in which you are located.

1. Ensure that the sponge inside the instrument's calibration chamber is wet. Insert the probe into the calibration chamber.



2. Turn the instrument on by pressing the **ON/OFF** button on the front of the instrument. Press the **MODE** button until dissolved oxygen is displayed in mg/L or %. Wait for the dissolved oxygen and temperature readings to stabilize (usually 15 minutes is required).

3. Use two fingers to press and release both the **UP ARROW** and **DOWN ARROW** buttons at the same time.

4. The LCD will prompt you to enter the local altitude in hundreds of feet. Use the arrow keys to increase or decrease the altitude. When the proper altitude appears on the LCD, press the **ENTER** button once.

EXAMPLE: Entering the number 12 here indicates 1200 feet.

5. The Model 85 should now display **CAL** in the lower left of the display, the calibration value should be displayed in the lower right of the display and the current % reading (before calibration) should be on the main display. Make sure that the current % reading (large display) is stable, then press the **ENTER** button. The display should read **SAVE** then should return to the Normal Operation Mode.

Each time the Model 85 is turned off, it may be necessary to re-calibrate before taking measurements. All calibrations should be completed at a temperature which is as close as possible to the sample temperature. Dissolved oxygen readings are only as good as the calibration.

5.2 CALIBRATION OF CONDUCTIVITY

IMPORTANT: System calibration is rarely required because of the factory calibration of the YSI Model 85. However, from time to time it is wise to check the system calibration and make adjustments when necessary.

Prior to calibration of the YSI Model 85, it is important to remember the following:

1. Always use clean, properly stored, NIST traceable calibration solutions (see Accessories and Replacement Parts). When filling a calibration container prior to performing the calibration procedures, make certain that the level of calibrant buffers is high enough in the container to cover the entire probe. Gently agitate the probe to remove any bubbles in the conductivity cell.
2. Rinse the probe with distilled water (and wipe dry) between changes of calibration solutions.
3. During calibration, allow the probe time to stabilize with regard to temperature (approximately 60 seconds) before proceeding with the calibration process. The readings after calibration are only as good as the calibration itself.
4. Perform sensor calibration at a temperature as close to 25°C as possible. This will minimize any temperature compensation error.

Follow these steps to perform an accurate calibration of the YSI Model 85:

1. Turn the instrument on and allow it to complete its self-test procedure.
2. Select a calibration solution that is most similar to the sample you will be measuring.
    - For sea water choose a 50 mS/cm conductivity standard (YSI Catalog# 3169)
    - For fresh water choose a 1 mS/cm conductivity standard (YSI Catalog# 3167)
    - For brackish water choose a 10 mS/cm conductivity standard (YSI Catalog # 3168)
3. Place at least 3 inches of solution in a clean glass beaker.
4. Use the **MODE** button to advance the instrument to display conductivity.
5. Insert the probe into the beaker deep enough so that the oval-shaped hole on the side of the probe is completely covered. Do not rest the probe on the bottom of the container -- suspend it above the bottom at least 1/4 inch.
6. Allow at least 60 seconds for the temperature reading to become stable.
7. Move the probe vigorously from side to side to dislodge any air bubbles from the electrodes.
8. Press and release the **UP ARROW** and **DOWN ARROW** buttons at the same time.

The **CAL** symbol will appear at the bottom left of the display to indicate that the instrument is now in Calibration mode.

9. Use the **UP ARROW** or **DOWN ARROW** button to adjust the reading on the display until it matches the value of the calibration solution you are using.
10. Once the display reads the exact value of the calibration solution being used (the instrument will make the appropriate compensation for temperature variation from 25°C), press the **ENTER** button once. The word "**SAVE"** will flash across the display for a second indicating that the calibration has been accepted.

The YSI Model 85 is designed to retain its last conductivity calibration permanently. Therefore, there is no need to calibrate the instrument after battery changes or power down.

SECTION 6 ADVANCED CONDUCTIVITY SETUP

The default settings of the YSI Model 85 are appropriate for the vast majority of measurement applications. However, some measurement applications require very specific measurement criteria. For that reason, we have made the YSI Model 85 flexible to accommodate these "advanced users."

If, for example, you are using the YSI Model 85 for a process control application that requires that the conductivity readings be compensated to 20 °C instead of 25 °C -- this is the section to read. Or, if your application for the YSI Model 85 involves the measurement of a very specific saline solution, the default temperature coefficient may need to be changed to get the very best measurement of that specific salt.

IMPORTANT: There is never a need to enter Advanced Setup Mode unless your special measurement application calls for a change in reference temperature and or temperature coefficient. Therefore, unless you are certain that your application requires a change to one or both of these criteria, do not modify the default reference temperature (25°C) or the default temperature coefficient (1.91%).

6.1 CHANGING THE TEMPERATURE COEFFICIENT

Follow these steps to modify the temperature coefficient of the Model 85.

1. Turn the instrument on and wait for it to complete its self-test procedure.
2. Use the **MODE** button to advance the instrument to display conductivity.
3. Press and release both the **DOWN ARROW** and the **MODE** buttons at the same time.

The **CAL** symbol will appear at the bottom left of the display. The large portion of the display will show **1.91 %** (or a value set previously using Advanced Setup).

4. Use the **UP ARROW** or **DOWN ARROW** button to change the value to the desired new temperature coefficient.
5. Press the **ENTER** button. The word "**SAVE**" will flash across the display for a second to indicate that your change has been accepted.
6. Press the **MODE** button to return to normal operation; the CAL symbol will disappear from the display.

6.2 CHANGING THE REFERENCE TEMPERATURE

Follow these steps to modify the reference temperature of the Model 85.

1. Turn the instrument on and wait for it to complete its self-test procedure.
2. Use the **MODE** button to advance the instrument to display conductivity.
3. Press and release both the **DOWN ARROW** and the **MODE** buttons at the same time.

The **CAL** symbol will appear at the bottom left of the display. The large portion of the display will show **1.91 %** (or a value set previously using Advanced Setup).

4. Press and release the **MODE** button; the large portion of the display will show **25.0C** (or a value set previously using Advanced Setup).
5. Use the **UP ARROW** or **DOWN ARROW** button to change the value to the desired new reference temperature (any value between 15 °C and 25 °C is acceptable).
6. Press the **ENTER** button. The word "**SAVE**" will flash across the display for a second to indicate that your change has been accepted.
7. The instrument will automatically return to normal operation mode.

6.3 CHANGING FROM AUTORANGING TO MANUAL RANGING

If your application is easier to perform using a manual range that you select, the YSI Model 85 allows you to turn off the default autoranging feature. While you are making conductivity or temperature compensated conductivity measurements, simply press and release the **UP ARROW** button. Each additional press of the **UP ARROW** button will cycle the Model 85 to a different manual range until you return again to autoranging. Five pushes of the **UP ARROW** button will cycle the Model 85 through the four manual ranges and return the instrument to autoranging.

NOTE: You may see an error message in some manual ranges if the manual range selected is not adequate for the sample you are measuring. If this happens, simply press and release the **UP ARROW** button again until a range is selected which is suitable for your sample. If you get lost and don't know if you're in a manual range or autoranging, simply turn the instrument off and back on. Also note that the conductivity units will flash while you are in manual range. The instrument will always default to autoranging when first turned on.

The four ranges of the YSI Model 85 are:

Range 1	Range 2	Range 3	Range 4
0 to 499.9 µS/cm	0 to 4999 µS/cm	0 to 49.99 mS/cm	0 to 200.0 mS/cm

SECTION 7 MAKING MEASUREMENTS

7.1 TURNING THE INSTRUMENT ON

Once the batteries are installed correctly, press the **ON/OFF** button. The instrument will activate all segments of the display for a few seconds, which will be followed by a self-test procedure that will last for several more seconds. During this power on self-test sequence, the instrument's microprocessor is verifying that the instrument is working properly. The Model 85 will display the cell constant of the conductivity probe when the self-test is complete. If the instrument were to detect an internal problem, the display would show a **continuous** error message. See the section entitled Troubleshooting for a list of these error messages.

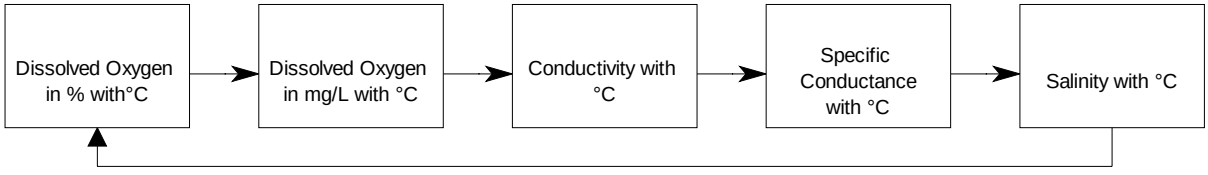
7.2 THE MEASUREMENT MODES OF THE MODEL 85

The Model 85 is designed to provide six distinct measurements:

- **Dissolved Oxygen %** -- A measurement of oxygen in percent of saturation.
- **Dissolved Oxygen mg/L** -- A measurement of oxygen in mg/L
- **Conductivity** -- A measurement of the conductive material in the liquid sample without regard to temperature
- **Specific Conductance** -- Also known as temperature compensated conductivity which automatically adjusts the reading to a calculated value which would have been read if the sample had been at 25^{o} C (or some other reference temperature which you choose). See Advanced Setup.
- **Temperature** -- which is always displayed.
- **Salinity** -- A calculation done by the instrument electronics, based upon the conductivity and temperature readings.

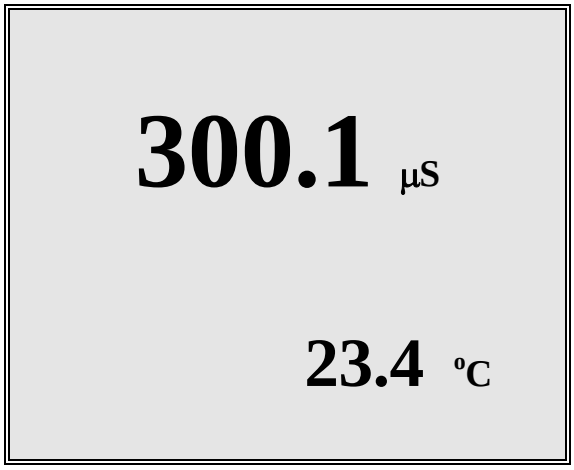
NOTE: When you turn the Model 85 off, it will "remember" which mode you used last and will return to that mode the next time the instrument is turned on.

To choose one of the measurement modes above (temperature is always displayed) simply press and release the **MODE** button. Carefully observe the small legends at the far right side of the LCD.



If the instrument is reading **Specific Conductance** the large numbers on the display will be followed by either a **µS** or an **mS.** Additionally the small portion of the display will show the **° C** flashing on and off.

If the instrument is reading **Conductivity** (not temperature compensated) the large numbers on the display will be followed by either a **µS** or an **mS.** Additionally the small portion of the display will show the **° C** **NOT** flashing.



If the instrument is reading **Dissolved Oxygen** the large numbers on the display will be followed by either a mg/L or %. It is important to remember that the dissolved oxygen probe is stirring dependent. This is due to the consumption of oxygen at the sensor tip during measurement. When taking dissolved oxygen measurements the probe must be moved through the sample at a rate of 1 foot per second to provide adequate stirring.

If the instrument is reading **Salinity** the large numbers on the display will be followed by a **ppt.**

7.3 AUTORANGING & RANGE SEARCHING

The YSI Model 85 is an autoranging instrument. This means that regardless of the conductivity or salinity of the solution (within the specifications of the instrument) all you need to do to get the most accurate reading is to put the probe in the sample. This feature makes the Model 85 as simple as possible to operate.

When you first place the Model 85 probe into a sample or calibration solution, and again when you first remove the probe the instrument will go into a range search mode that may take as long as 5 seconds. During some range searches the instrument display will flash **rANG** to indicate its movement from one range to another. The length of the range search depends on the number of ranges that must be searched in order to find the correct range for the sample. During the range search, the instrument will appear to freeze on a given reading for a few seconds then, once the range is located, will pinpoint the exact reading on the display. The display may also switch to **00.0** for a second or two during a range search before it selects the proper range.

7.4 THE BACKLIGHT

At times it may be necessary to take measurements with the Model 85 in dark or poorly lit areas. To help in this situation, the Model 85 comes equipped with a backlight that will illuminate the display so that it can be easily read. To activate the backlight, press and hold the **LIGHT** button. The display will remain lit as long as the button is depressed. When you release it, the light goes out to preserve battery life.

SECTION 8 SAVING DATA

The Model 85 is equipped with non-volatile memory that is capable of storing up to 50 different sets of readings. Non-volatile means that you do not need to worry that your data will be lost due to a power failure or power interrupt. The Model 85 will also assign a site identity number to each set of readings to allow easy review of the data. This feature is useful in situations where transcribing data is difficult or not available.

8.1 SAVING DATA TO MEMORY

1. While any parameter is displayed on the screen depress the **ENTER** button and hold for approximately 2 seconds. The meter will flash **SAVE** on the display along with the current site identity being used.

2. When all 50 sites are full the display will flash **FULL** on the screen. This message will remain on the screen (even after power down) until a button is pushed.

Once you have acknowledged the memory is full, any subsequent saved data will begin overwriting existing data starting with site #1.

8.2 RECALLING STORED DATA

1. To put the Model 85 into the **RECALL** mode depress the **MODE** button repeatedly until **rcl** is displayed on the screen along with the site ID number in the lower right corner. (see figure #1)

2. Depress the **ENTER** button to review the last set of data that was saved. The Model 85 will display the dissolved oxygen in % saturation and temperature. Another press of the **ENTER** button will display the dissolved oxygen in mg/L and the temperature.

Depress the **ENTER** button again and again to review the conductivity, specific conductivity and salinity readings. All of which are displayed with the temperature.

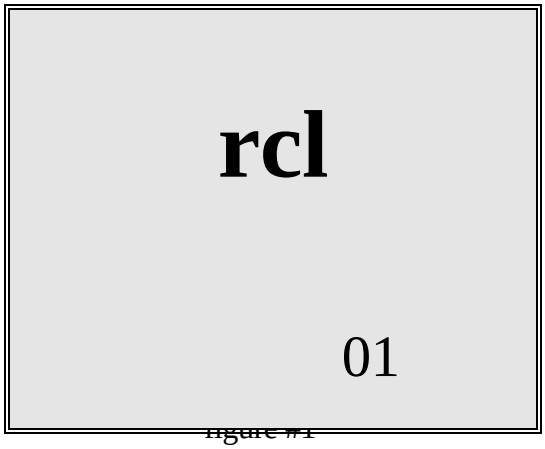


Figure #1

3. Depress the **UP ARROW** button to increment through the saved sets of data.

4. Depress the **DOWN ARROW** button to decrement through the saved sets of data.

5. When the correct site ID# is displayed, press the **ENTER** button to display the data.

6. When you have finished recalling data, press the **MODE** button to return to normal operation.

NOTE: The Model 85 will recall data as a list. When the **UP ARROW** is depressed the Model 85 will display the Site ID# for the previously recorded date. For example: If you are reviewing Site ID# 5 and the **UP ARROW** is depressed the Model 85 will display Site ID# 4. If you are reviewing Site ID# 5 and Site ID# 5 was the last set of data stored the **DOWN ARROW** button will display Site ID# 1.

Here is an example of the Model 85 memory.
Site ID #1
Site ID #2
Site ID #3 ←—If the **UP ARROW** button was pressed the Model 85 would display Site ID #2
Site ID #4
Site ID #5

8.3 ERASING STORED DATA

1. To erase the data that is stored into the Model 85's memory, depress the **MODE** button repeatedly until the Model 85 displays **ErAS** on the screen. (see figure #2)

2. Depress and hold the **DOWN ARROW** and **ENTER** buttons simultaneously for approximately 5 seconds.

3. The Model 85 flashing DONE on the display for 1 to 2 seconds indicates successful erasure. The instrument will automatically change to normal operation after completion.

IMPORTANT: Data in all 50 site ID's will be erased completely and will be lost forever. Do not use the erase function until all recorded data has been transcribed to an archive outside the Model 85.

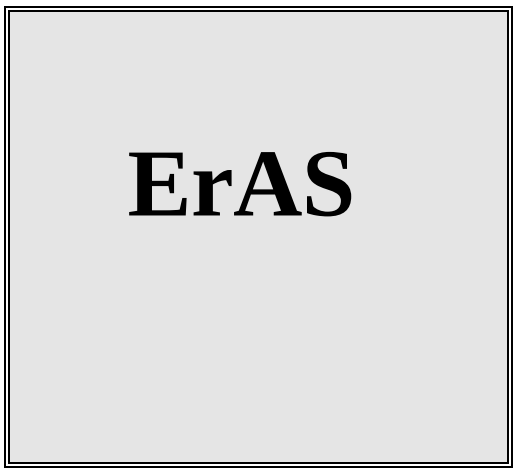


figure #2

SECTION 9 MAINTENANCE

9.1 CLEANING AND STORAGE

The single most important requirement for accurate and reproducible results in conductivity measurement is a clean cell. A dirty cell will change the conductivity of a solution by contaminating it.

NOTE: ALWAYS RINSE THE CONDUCTIVITY CELL WITH CLEAN WATER AFTER EACH USE.

To clean the conductivity cell:

1. Dip the cell in cleaning solution and agitate for two to three minutes. Any one of the foaming acid tile cleaners, such as Dow Chemical Bathroom Cleaner, will clean the cell adequately. When a stronger cleaning preparation is required, use a solution of 1:1 isopropyl alcohol and 1 N HCl. Remove the cell from the cleaning solution.

2. Use the nylon brush (supplied) to dislodge any contaminants from inside the electrode chamber.

3. Repeat steps one and two until the cell is completely clean. Rinse the cell thoroughly in deionized, or clean tap water.

4. Store the conductivity cell in the meter storage chamber.

NOTE: See Section 11, Dissolved Oxygen Probe Precautions for instructions on cleaning the dissolved oxygen electrodes.

SECTION 10 PRINCIPLES OF OPERATION

The dissolved oxygen sensor utilizes an oxygen permeable membrane that covers an electrolytic cell consisting of a gold cathode and a porous silver anode. This membrane acts as a diffusion barrier and an isolation barrier preventing fouling of the cathode surface by impurities in the environment. Upon entering the cell through the membrane, oxygen is reduced at an applied potential of -0.8 V referenced to the silver electrode. The reduction current at the cathode is directly proportional to the partial pressure of oxygen in liquid (expressed as %-air saturation) which is proportional to the concentration of dissolved oxygen (in mg/L) at a particular temperature. Thus the same partial pressure of oxygen (% air-saturation) in liquid gives different concentrations of dissolved oxygen (mg/L) at different temperatures because of the different solubility's of oxygen at different temperatures.

The conductivity cell utilizes four pure nickel electrodes for the measurement of solution conductance. Two of the electrodes are current driven, and two are used to measure the voltage drop. The measured voltage drop is then converted into a conductance value in milli-Siemens (millimhos). To convert this value to a conductivity (specific conductance) value in milli-Siemens per cm (mS/cm), the conductance is multiplied by the cell constant that has units of reciprocal cm (cm^{-1}). The cell constant for the Model 85 conductivity cell is 5.0/cm $\pm$ 4%. For most applications, the cell constant is automatically determined (or confirmed) with each deployment of the system when the calibration procedure is followed. Solutions with conductivity's of 1.00, 10.0, 50.0, and 100.0 mS/cm, which have been prepared in accordance with recommendation 56-1981 of the Organisation Internationale de Métrologie Légale (OIML) are available from YSI. The instrument output is in µS/cm or mS/cm for both conductivity and specific conductance. The multiplication of cell constant times conductance is carried out automatically by the software.

10.1 TEMPERATURE EFFECT ON CONDUCTIVITY

The conductivity of solutions of ionic species is highly dependent on temperature, varying as much as 3% for each change of one degree Celsius (temperature coefficient = 3%/C). In addition, the temperature coefficient itself varies with the nature of the ionic species present.

Because the exact composition of a natural media is usually not known, it is best to report a conductivity at a particular temperature, e.g. 20.2 mS/cm at 14 C. However, in many cases, it is also useful to compensate for the temperature dependence in order to determine at a glance if gross changes are occurring in the ionic content of the medium over time. For this reason, the Model 85 software also allows the user to output conductivity data in either raw or temperature compensated form. If "Conductivity" is selected, values of conductivity that are NOT compensated for temperature are output to the display. If "Specific Conductance" is selected, the Model 85 uses the temperature and raw conductivity values associated with each determination to generate a specific conductance value compensated to a user selected reference temperature (see Advanced Setup) between 15 C and 25 C. Additionally the user can select any temperature coefficient from 0% to 4% (see Advanced Setup). Using the Model 85 default reference temperature and temperature coefficient (25 C and 1.91%), the calculation is carried out as in equation (1) below:

$$\text{Specific Conductance (25°C)} = \frac{\text{Conductivity}}{1 + TC * (T - 25)}$$

As noted above, unless the solution being measured consists of pure KCl in water, this temperature compensated value will be somewhat inaccurate, but the equation with a value of TC = 0.0191 will provide a close approximation for solutions of many common salts such as NaCl and NH_4Cl and for seawater.

Salinity is determined automatically from the Model 85 conductivity readings according to algorithms found in Standard Methods for the Examination of Water and Wastewater (ed. 1989). The use of the Practical Salinity Scale 1978 results in values which are unitless, since the measurements are carried out in reference to the conductivity of standard seawater at 15 C. However, the unitless salinity values are very close to those determined by the previously-used method where the mass of dissolved salts in a given mass of water (parts per thousand) was reported. Hence, the designation "ppt" is reported by the instrument to provide a more conventional output. For further information on conductivity and the above standard information, refer to the ASTM document, Standard Methods of Test for Electrical Conductivity of Water and Industrial Wastewater, ASTM Designation D1125-82, and OIML Recommendation Number 56. ASTM symbols for conductivity, cell constant, and path length differ from those preferred in the general literature and also from those used in this manual.

SECTION 11 DISCUSSION OF MEASUREMENT ERRORS

11.1 DISSOLVED OXYGEN MEASUREMENT ERRORS

There are three basic types of error. Type 1 errors are related to limitations of instrument design and tolerances of instrument components. These are chiefly the meter linearity and the resistor tolerances. Type 2 errors are due to basic probe accuracy tolerances, chiefly background signal, probe linearity, and variations in membrane temperature coefficient. Type 3 errors are related to the operator's ability to determine the conditions at the time of calibration. If calibration is performed against more accurately known conditions, type 3 errors are appropriately reduced.

The sample calculations that follow are for a near extreme set of conditions.

TYPE 1 ERRORS

A. Meter linearity error: ±1% of full scale reading, or ±0.15 mg/l

B. Component and circuitry error: ±0.05 mg/l

TYPE 2 ERRORS

A. Temperature compensation for membrane temperature coefficient: ±0.03 mg/l

B. Temperature measurement errors: A maximum ±0.2^{o}C probe error is equal to ±0.14 mg/l

TYPE 3 ERRORS

A. Altitude:

A 1000-foot change in altitude is equal to an error of approximately 3% at the 10 mg/l level.

B. Humidity:

Errors occur if calibration is performed at less than 100% humidity. The error varies with the temperature as follows:

TEMPERATURE	ERROR
0°C	0.02 mg/l
10°C	0.05 mg/l
20°C	0.12 mg/l
30°C	0.27 mg/l
40°C	0.68 mg/l

APPROXIMATING THE ERROR

It is unlikely that the actual error in any measurement will be the maximum possible error. A better error approximation is obtained using a root mean squared (r.m.s.) calculation:

$$\text{r.m.s. error} = \pm[1a^2 + 1b^2 + 2a^2 + 2b^2 + 3a^2 + 3b^2]^{½} \text{ mg/l}$$

11.2 CONDUCTIVITY MEASUREMENT ERRORS

System accuracy for conductivity measurements is equal to the sum of the errors contributed by the environment and the various components of the measurement setup. These include:

- Instrument accuracy
- Cell-constant error
- Solution temperature offset
- Cell contamination (including air bubbles)
- Electrical noise
- Galvanic effects

Only the first three are of major concern for typical measurements, although the user should also be careful to see that cells are clean and maintained in good condition at all times.

Instrument Accuracy = ± .5% maximum
The accuracy specified for the range being used is the worst case instrument error.

Cell-Constant Error = ± .5% maximum
Although YSI cells are warranted to be accurate to within one percent, you should still determine the exact cell constant of your particular cell. Contamination or physical damage to the cell can alter the cell constant. Performing a calibration will eliminate any error that might arise because of cell constant change.

YSI cells are calibrated to within one percent of the stated cell constant at a single point. We consider these products to be usefully linear over most instrument ranges. The cell constant can be calibrated to ±0.35% accuracy with YSI conductivity calibrator solutions.

Temperature Error = ± 1% maximum
The solution temperature error is the product of the temperature coefficient and the temperature offset from 25 C, expressed as a percentage of the reading that would have been obtained at 25 C. The error is not necessarily a linear function of temperature. The statement of error is derived from a 25 C temperature offset and a 3%/ C temperature coefficient.

Total Error
Considering only the above three factors, system accuracy under worst case conditions will be ±2%, although the actual error will be considerably less if recommended and properly calibrated cells and instrument ranges are used. Additional errors, which can essentially be eliminated with proper handling, are described below.

Cell Contamination
This error is usually due to contamination of the solution being measured, which occurs when solution is carried-over from the last solution measured. Thus, the instrument might be correctly reporting the conductivity seen, but the reading does not accurately represent the value of the bulk

solution. Errors will be most serious when low conductivity solutions are contaminated by carry-over from high conductivity solutions, and can then be of an order of magnitude or more.

Follow the cleaning instructions carefully before attempting low conductivity measurements with a cell of unknown history or one that has been previously used in higher value solutions.

An entirely different form of contamination sometimes occurs due to a buildup of foreign material directly on cell electrodes. While rare, such deposits have, on occasion, markedly reduced the effectiveness of the electrodes. The result is an erroneously low conductance reading.

Electrical-Noise Errors

Electrical noise can be a problem in any measurement range, but will contribute the most error and be the most difficult to eliminate when operating in the lowest ranges. The noise may be either line-conducted or radiated or both, and may require, grounding, shielding, or both.

Galvanic and Miscellaneous Effects

In addition to the error sources described above, there is another class of contributors that can be ignored for all but the most meticulous of laboratory measurements. These errors are always small and are generally completely masked by the error budget for cell-constant calibration, instrument accuracy, etc. Examples range from parasitic reactance associated with the solution container and its proximity to external objects to the minor galvanic effects resulting from oxide formation or deposition on electrodes. Only trial and error in the actual measurement environment can be suggested as an approach to reduce such errors. If the reading does not change as the setup is adjusted, errors due to such factors can be considered too small to see.

11.3 DISSOLVED OXYGEN PROBE PRECAUTIONS

1. Membrane life depends on usage. Membranes will last a long time if installed properly and treated with care. Erratic readings are a result of loose, wrinkled, damaged, or fouled membranes, or from large (more than 1/8" diameter) bubbles in the electrolyte reservoir. If erratic readings or evidence of membrane damage occurs, you should replace the membrane and the KCl solution. The average replacement interval is two to four weeks.

2. If the membrane is coated with oxygen consuming (e.g. bacteria) or oxygen evolving organisms (e.g. algae), erroneous readings may occur.

3. Chlorine, sulfur dioxide, nitric oxide, and nitrous oxide can affect readings by behaving like oxygen at the probe. If you suspect erroneous readings, it may be necessary to determine if these gases are the cause.

4. Avoid any environment that contains substances that may attack the probe materials. Some of these substances are concentrated acids, caustics, and strong solvents. The probe materials that come in contact with the sample include FEP Teflon, stainless steel, epoxy, polyetherimide and the polyurethane cable covering.

5. For correct probe operation, the gold cathode must always be bright. If it is tarnished (which can result from contact with certain gases) or plated with silver, the gold surface must be restored. To restore the cathode, you may either return the instrument to the factory or clean it using the YSI 5238 probe reconditioning kit. Never use chemicals or abrasives not supplied with this kit.

NOTE: Model 85 probes built before July, 1996 (serial numbers starting with 96F or lower), should be cleaned with the sanding disc mounted on a FLAT surface. Do NOT use the curved tool provided in the 5238 probe reconditioning kit on these probes.

6. It is also possible for the silver anode to become contaminated, which will prevent successful calibration. To clean the anode, remove the membrane and soak the probe overnight in 3% ammonium hydroxide. Next, rinse the sensor tip with deionized water, add new KCl solution, and install a new membrane. Turn the instrument on and allow the system to stabilize for at least 30 minutes. If, after several hours, you are still unable to calibrate, return the YSI Model 85 system to an authorized service center for service.

7. To keep the electrolyte from drying out, store the probe in the calibration chamber with the small piece of sponge.

SECTION 12 TROUBLESHOOTING

SYMPTOM	POSSIBLE CAUSE	ACTION
1. Instrument will not turn on	A. Low battery voltage B. Batteries installed wrong C. Meter requires service	A. Replace batteries B. Check battery polarity. C. Return system for service
2. Instrument will not calibrate (Dissolved Oxygen)	A. Membrane is fouled or damaged B. Probe anode is fouled or dark C. Probe cathode is tarnished D. System requires service	A. Replace membrane & KCl B. Clean anode C. Clean cathode D. Return system for service
3. Instrument will not calibrate (Conductivity)	A. Cell is contaminated	A. See "Maintenance" Section
4. Instrument "locks up"	A. Instrument has rec'd a shock B. Batteries are low or damaged C. System requires service	A & B. Remove battery lid, wait 15 seconds for reset, replace lid. C. Return system for service
5. Instrument readings are inaccurate (Dissolved Oxygen)	A. Cal altitude is incorrect B. Probe not in 100% O_2 saturated air during Cal procedure C. Membrane fouled or damaged D. Probe anode is fouled or dark E. Probe cathode is tarnished F. System requires service	A. Recalibrate w/correct value B. Moisten sponge & place in Cal chamber w/ probe & Recal C. Replace membrane D. Clean anode E. Clean cathode F. Return system for service
6. Instrument readings are inaccurate (Conductivity)	A. Calibration is required B. Cell is contaminated C. Tempco is set incorrectly D. Reference temperature incorrect E. Readings are or are not temperature compensated.	A. See "Calibration" Section B. See "Maintenance" Section C. See "Advanced Setup" Section D. See "Advanced Setup" Section E. See "Making Measurements" Section
7..LCD displays "LO BAT" Main display flashes "off"	A. Batteries are low or damaged	A. Replace batteries
8. Main Display reads "OVEr" (Secondary display reads "ovr") (Secondary display reads "udr")	A. Conductivity reading is >200 mS B. Temperature reading is >65°C C. Temperature reading is <-5°C D. Salinity reading is >80 ppt E. User cell constant cal K is >5.25 F. DO temperature is >46°C G. DO % saturation is >200% H. DO concentration is >20 mg/L	In all cases, check calibration values and procedures; check advanced setup settings. If each of these are set correctly, return instrument for service.
9. Main display reads "Undr"	A. User cell constant cal K is <4.9 B. DO current too low to calibrate	A. Recalibrate instrument using known good conductivity standard. Follow cell cleaning procedure in the Maintenance section. B. Replace membrane, clean probe
10. Main display reads "rErr"	A. Reading exceeds user selected manual range.	A. Use the mode key to select a higher or lower manual range, or set system to autoranging.
11. Main display reads "PErr"	A. User cell constant cal K is 0.0 B. Incorrect sequence of keystrokes.	A. See "Advanced Setup" section. B. Refer to manual section for step by step instruction for the function you are attempting.

SYMPTOM	POSSIBLE CAUSE	ACTION
12. Main display reads “LErr”	A. In temperature compensated conductivity mode, temperature exceeds the values computed using user defined temperature coefficient and/or reference temperature. B. In cell constant cal mode, temperature exceeds the values computed using user defined temperature coefficient and/or reference temperature.	A. & B. Adjust user defined tempco or reference temperature. (pg. 10)
13. Main display reads “Err” (Secondary display reads “ra”)	A. System has failed its RAM test check procedure.	A. Turn instrument OFF and back ON again. B. Return the system for service (pg. 26)
14. Main display reads “Err” (Secondary display reads “ro”)	A. System has failed its ROM test check procedure.	A. Turn instrument OFF and back ON again. B. Return the system for service (pg. 26)
15. Secondary display reads “rEr”	A. Temperature jumper is set to °F and reading is >199.9°F but <203°F.	A. Return the system for service. (pg. 26)
16. Main display reads “FAIL” (Secondary display reads “eep”)	A. EEPROM has failed to respond in time.	A. Return the system for service. (pg. 26)
17. Readings on main display don’t change	A. Meter is in recall mode.	A. Press **MODE** button to return to Normal Operation (pg. 12)

SECTION 13 WARRANTY AND REPAIR

YSI Model 85 Handheld Meters are warranted for two years from date of purchase by the end user against defects in materials and workmanship. YSI Model 85 probes and cables are warranted for one year from date of purchase by the end user against defects in material and workmanship. Within the warranty period, YSI will repair or replace, at its sole discretion, free of charge, any product that YSI determines to be covered by this warranty.

To exercise this warranty, write or call your local YSI representative, or contact YSI Customer Service in Yellow Springs, Ohio. Send the product and proof of purchase, transportation prepaid, to the Authorized Service Center selected by YSI. Repair or replacement will be made and the product returned, transportation prepaid. Repaired or replaced products are warranted for the balance of the original warranty period, or at least 90 days from date of repair or replacement.

Limitation of Warranty

This Warranty does not apply to any YSI product damage or failure caused by (i) failure to install, operate or use the product in accordance with YSI's written instructions, (ii) abuse or misuse of the product, (iii) failure to maintain the product in accordance with YSI's written instructions or standard industry procedure, (iv) any improper repairs to the product, (v) use by you of defective or improper components or parts in servicing or repairing the product, or (vi) modification of the product in any way not expressly authorized by YSI.

THIS WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. YSI's LIABILITY UNDER THIS WARRANTY IS LIMITED TO REPAIR OR REPLACEMENT OF THE PRODUCT, AND THIS SHALL BE YOUR SOLE AND EXCLUSIVE REMEDY FOR ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY. IN NO EVENT SHALL YSI BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY.

AUTHORIZED U.S. SERVICE CENTERS

Please visit www.ysi.com or contact YSI Technical Support for the nearest authorized service center.

YSI Incorporated • Technical Support • Phone: +1 937 767-7241 • 800 897-4151 • Fax: 937 767-1058 • Email: environmental@ysi.com

CLEANING INSTRUCTIONS

NOTE: Before they can be serviced, equipment exposed to biological, radioactive, or toxic materials must be cleaned and disinfected. Biological contamination is presumed for any instrument, probe, or other device that has been used with body fluids or tissues, or with wastewater. Radioactive contamination is presumed for any instrument, probe or other device that has been used near any radioactive source.

If an instrument, probe, or other part is returned or presented for service without a Cleaning Certificate, and if in our opinion it represents a potential biological or radioactive hazard, our service personnel reserve the right to withhold service until appropriate cleaning, decontamination, and certification has been completed. We will contact the sender for instructions as to the disposition of the equipment. Disposition costs will be the responsibility of the sender.

When service is required, either at the user's facility or at YSI, the following steps must be taken to insure the safety of our service personnel.

1. In a manner appropriate to each device, decontaminate all exposed surfaces, including any containers. 70% isopropyl alcohol or a solution of 1/4 cup bleach to 1-gallon tap water are suitable for most disinfecting. Instruments used with wastewater may be disinfected with .5% Lysol if this is more convenient to the user.
2. The user shall take normal precautions to prevent radioactive contamination and must use appropriate decontamination procedures should exposure occur.
3. If exposure has occurred, the customer must certify that decontamination has been accomplished and that no radioactivity is detectable by survey equipment.
4. Any product being returned to the YSI Repair Center, should be packed securely to prevent damage.
5. Cleaning must be completed and certified on any product before returning it to YSI.

PACKING INSTRUCTIONS

1. Clean and decontaminate items to insure the safety of the handler.
2. Complete and include the Cleaning Certificate.
3. Place the product in a plastic bag to keep out dirt and packing material.
4. Use a large carton, preferably the original, and surround the product completely with packing material.
5. Insure for the replacement value of the product.

Cleaning Certificate

Organization _____

Department _____

Address _____

City _____ State _ Zip _____

Country _____ Phone _____

Model No. of Device _ Lot Number _____

Contaminant (if known) _____

Cleaning Agent(s) used _____

Radioactive Decontamination Certified?

(Answer only if there has been radioactive exposure)

_____ Yes _____ No

Cleaning Certified By _____

Name Date

SECTION 14 ACCESSORIES AND REPLACEMENT PARTS

The following parts and accessories are available from YSI or any Franchise Dealer authorized by YSI.

YSI ORDER NUMBER	DESCRIPTION
YSI 5906	Replacement Membrane Cap Kit (6 each)
YSI 5238	Probe Reconditioning Kit
YSI 3161	Conductivity Calibration Solution 1,000 µ/cm (1 Quart)
YSI 3163	Conductivity Calibration Solution 10,000 µ/cm (1 Quart)
YSI 3165	Conductivity Calibration Solution 100,000 µ/cm (1 Quart)
YSI 3167	Conductivity Calibration Solution 1,000 µ/cm (8 pints)
YSI 3168	Conductivity Calibration Solution 10,000 µ/cm (8 pints)
YSI 3169	Conductivity Calibration Solution 50,000 µ/cm (8 pints)
YSI 5520	Carrying Case
YSI 118510	Replacement Probe & Cable Assembly (10 feet)
YSI 118522	Replacement Probe & Cable Assembly (25 feet)
YSI 118527	Replacement Probe & Cable Assembly (50 feet)
YSI 118519	Replacement Probe and Cable Assembly (100 feet)
YSI 038501	Replacement Front Case Cover
YSI 055242	Replacement Rear Case Cover
YSI 055244	Replacement Battery Cover Kit
YSI 055204	Replacement Case Gasket and Screw
YSI 055219	Storage Chamber Sponge
YSI 030156	Main Board Assembly
YSI 038213	Replacement Electrode Cleaning Brush

APPENDIX A SPECIFICATIONS

Operating Environment

Medium: fresh, sea, or polluted water and most other liquid solutions.
Temperature: -5 to +65 °C
Depth: 0 to 10, 0 to 25, 0 to 50, or 0 to 100 feet (depending on cable length)

Storage Temperature: -10 to +50 °C

Material: ABS, Stainless Steel, and other materials

Dimensions:

Height:	9.5 inches	(24.13 cm)
Thickness:	2.2 inches	(5.6 cm)
Width:	3.5 inches max.	(8.89 cm)
Weight:	1.7 pounds (w/ 10' cable)	(.77 kg)
Display:	2.3"W x 1.5"L	(5.8cm W x 3.8cm L)

Power: 9 VDC -6 AA-size Alkaline Batteries (included)
Approximately 100 hours operation from each new set of batteries

Water Tightness: Meets or exceeds IP65 standards

Extensive testing of the YSI Model 85 indicates the following typical performance:

Measurement	Range	Resolution	Accuracy
Conductivity	0 to 499.9 µS/cm	0.1 µS/cm	± .5% FS
	0 to 4999 µS/cm	1.0 µS/cm	± .5% FS
	0 to 49.99 mS/cm	.01 mS/cm	± .5% FS
	0 to 200.0 mS/cm	0.1 mS/cm	± .5% FS
Salinity	0 to 80 ppt	.1 ppt	± 2%, or ± 0.1 ppt
Temperature	-5 to +65 °C	0.1 °C	± 0.1 °C (±1 lsd)
Dissolved Oxygen	0 to 200 % Air Sat.	0.1% Air Saturation	± 2% Air Saturation
	0 to 20 mg/L	0.01 mg/L	± 0.3 mg/L

Adjustable Conductivity Reference Temperature: 15°C to 25°C

Adjustable Temperature Compensation Factor for Conductivity: 0% to 4%

Temperature Compensation: Automatic

Range: Autoranging for Dissolved Oxygen

User selected or Autoranging for Conductivity

APPENDIX B - TEMPERATURE CORRECTION DATA

Temperature Correction Data for Typical Solutions

A. Potassium Chloride** (KCl)

Concentration: 1 mole/liter			Concentration: 1×10^{-1} mole/liter		
C	mS/cm	%/ C (to 25 C)	C	mS/cm	%/ C (to 25 C)
0	65.10	1.67	0	7.13	1.78
5	73.89	1.70	5	8.22	1.80
10	82.97	1.72	10	9.34	1.83
15	92.33	1.75	15	10.48	1.85
20	101.97	1.77	20	11.65	1.88
25	111.90	1.80	25	12.86	1.90
			30	14.10	1.93
			35	15.38	1.96
			37.5	16.04	1.98
			40	16.70	1.99
			45	18.05	2.02
			50	19.43	2.04

Concentration: 1×10^{-2} mole/liter			Concentration: 1×10^{-3} mole/liter		
C	mS/cm	%/ C (to 25 C)	C	mS/cm	%/ C (to 25 C)
0	0.773	1.81	0	0.080	1.84
5	0.892	1.84	5	0.092	1.88
10	1.015	1.87	10	0.105	1.92
15	1.143	1.90	15	0.119	1.96
20	1.275	1.93	20	0.133	1.99
25	1.412	1.96	25	0.147	2.02
30	1.553	1.99	30	0.162	2.05
35	1.697	2.02	35	0.178	2.07
37.5	1.771	2.03	37.5	0.186	2.08
40	1.845	2.05	40	0.194	2.09
45	1.997	2.07	45	0.210	2.11
50	2.151	2.09	50	0.226	2.13

** Charts developed by interpolating data from International Critical Tables, Vol. 6, pp. 229-253, McGraw-Hill Book Co., NY.

B. Sodium Chloride* (NaCl)

Saturated solutions at all temperatures			Concentration: 0.5 mole/liter		
C	mS/cm	%/ C (to 25 C)	C	mS/cm	%/ C (to 25 C)
0	134.50	1.86	0	25.90	1.78
5	155.55	1.91	5	29.64	1.82
10	177.90	1.95	10	33.61	1.86
15	201.40	1.99	15	37.79	1.90
20	225.92	2.02	20	42.14	1.93
25	251.30	2.05	25	46.65	1.96
30	277.40	2.08	30	51.28	1.99
			35	56.01	2.01
			37.5	58.40	2.02
			40	60.81	2.02
			45	65.65	2.04
			50	70.50	2.05

Concentration: 1 x 10^{-1} mole/liter			Concentration: 1 x 10^{-2} mole/liter		
C	mS/cm	%/ C (to 25 C)	C	mS/cm	%/ C (to 25 C)
0	5.77	1.83	0	0.632	1.87
5	6.65	1.88	5	0.731	1.92
10	7.58	1.92	10	0.836	1.97
15	8.57	1.96	15	0.948	2.01
20	9.60	1.99	20	1.064	2.05
25	10.66	2.02	25	1.186	2.09
30	11.75	2.04	30	1.312	2.12
35	12.86	2.06	35	1.442	2.16
37.5	13.42	2.07	37.5	1.508	2.17
40	13.99	2.08	40	1.575	2.19
45	15.14	2.10	45	1.711	2.21
50	16.30	2.12	50	1.850	2.24

Concentration: 1 x 10^{-3} mole/liter		
C	mS/cm	%/ C (to 25 C)
0	0.066	1.88
5	0.076	1.93
10	0.087	1.98
15	0.099	2.02
20	0.111	2.07
25	0.124	2.11
30	0.137	2.15
35	0.151	2.19
37.5	0.158	2.20
40	0.165	2.22
45	0.180	2.25
50	0.195	2.29

* Charts developed by interpolating data from the CRC Handbook of Chemistry and Physics, 42nd ed., p. 2606, The Chemical Rubber Company, Cleveland.

C. Lithium Chloride* (LiCl)

Concentration: 1 mole/liter			Concentration: 1 x 10^{-1} mole/liter		
C	mS/cm	%/ C (to 25 C)	C	mS/cm	%/ C (to 25 C)
0	39.85	1.82	0	5.07	1.87
5	46.01	1.85	5	5.98	1.85
10	52.42	1.89	10	6.87	1.85
15	59.07	1.92	15	7.75	1.85
20	65.97	1.95	20	8.62	1.85
25	73.10	1.98	25	9.50	1.86
30	80.47	2.02	30	10.40	1.88
35	88.08	2.05	35	11.31	1.91
37.5	91.97	2.07	37.5	11.78	1.92
40	95.92	2.08	40	12.26	1.94
45	103.99	2.11	45	13.26	1.98
50	112.30	2.15	50	14.30	2.02

Concentration: 1 x 10^{-2} mole/liter			Concentration: 1 x 10^{-3} mole/liter		
C	mS/cm	%/ C (to 25 C)	C	mS/cm	%/ C (to 25 C)
0	0.567	1.88	0	0.059	1.93
5	0.659	1.92	5	0.068	2.03
10	0.755	1.96	10	0.078	2.12
15	0.856	2.00	15	0.089	2.19
20	0.961	2.04	20	0.101	2.25
25	1.070	2.08	25	0.114	2.28
30	1.183	2.12	30	0.127	2.31
35	1.301	2.16	35	0.140	2.32
37.5	1.362	2.18	37.5	0.147	2.32
40	1.423	2.20	40	0.154	2.31
45	1.549	2.24	45	0.166	2.29
50	1.680	2.28	50	0.178	2.25

D. Potassium Nitrate** (KNO_3)

Concentration: 1 x 10^{-1} mole/liter			Concentration: 1 x 10^{-2} mole/liter		
C	mS/cm	%/ C (to 25 C)	C	mS/cm	%/ C (to 25 C)
0	6.68	1.78	0	0.756	1.77
5	7.71	1.79	5	0.868	1.80
10	8.75	1.81	10	0.984	1.83
15	9.81	1.83	15	1.105	1.86
20	10.90	1.85	20	1.229	1.88
25	12.01	1.87	25	1.357	1.90
30	13.15	1.90	30	1.488	1.93
35	14.32	1.92	35	1.622	1.95
37.5	14.92	1.94	37.5	1.690	1.96
40	15.52	1.95	40	1.759	1.97
45	16.75	1.97	45	1.898	1.99
50	18.00	2.00	50	2.040	2.01

* Charts developed by interpolating data from the CRC Handbook of Chemistry and Physics, 42nd ed., p. 2606, The Chemical Rubber Company, Cleveland.
** Charts developed by interpolating data from International Critical Tables, Vol. 6, pp. 229-253, McGraw-Hill Book Co., NY.

E. Ammonium Chloride* (NH_4Cl)

Concentration: 1 mole/liter			Concentration: 1×10^{-1} mole/liter		
C	mS/cm	%/ C (to 25 C)	C	mS/cm	%/ C (to 25 C)
0	64.10	1.60	0	6.96	1.82
5	74.36	1.53	5	7.98	1.88
10	83.77	1.45	10	9.09	1.93
15	92.35	1.37	15	10.27	1.97
20	100.10	1.29	20	11.50	2.00
25	107.00	1.21	25	12.78	2.03
			30	14.09	2.06
			35	15.43	2.07
			37.5	16.10	2.08
			40	16.78	2.08
			45	18.12	2.09
			50	19.45	2.09

Concentration: 1×10^{-2} mole/liter			Concentration: 1×10^{-3} mole/liter		
C	mS/cm	%/ C (to 25 C)	C	mS/cm	%/ C (to 25 C)
0	0.764	1.84	0	0.078	1.88
5	0.889	1.86	5	0.092	1.90
10	1.015	1.88	10	0.105	1.91
15	1.144	1.91	15	0.119	1.93
20	1.277	1.94	20	0.133	1.95
25	1.414	1.97	25	0.148	1.98
30	1.557	2.02	30	0.162	2.01
35	1.706	2.06	35	0.178	2.04
37.5	1.782	2.08	37.5	0.186	2.06
40	1.860	2.10	40	0.194	2.07
45	2.020	2.14	45	0.210	2.11
50	2.186	2.18	50	0.227	2.15

* Charts developed by interpolating data from the CRC Handbook of Chemistry and Physics, 42nd ed., p. 2606, The Chemical Rubber Company, Cleveland.

APPENDIX C REQUIRED NOTICE

The Federal Communications Commission defines this product as a computing device and requires the following notice:

This equipment generates and uses radio frequency energy and if not installed and used properly, may cause interference to radio and television reception. There is no guarantee that interference will not occur in a particular installation. If this equipment does cause interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- re-orient the receiving antenna
- relocate the computer with respect to the receiver
- move the computer away from the receiver
- plug the computer into a different outlet so that the computer and receiver are on different branch circuits.

If necessary, the user should consult the dealer or an experienced radio/television technician for additional suggestions. The user may find the following booklet, prepared by the Federal Communications Commission, helpful: "How to Identify and Resolve Radio-TV Interference Problems." This booklet is available from the U.S. Government Printing Office, Washington, D.C. 20402, Stock No. 0004-000-00345-4.

APPENDIX D CONVERSION CHART

TO CONVERT FROM	TO	EQUATION
Feet	Meters	Multiply by 0.3048
Meters	Feet	Multiply by 3.2808399
Degrees Celsius	Degrees Fahrenheit	(9/5 ^{o}C)+32
Degrees Fahrenheit	Degrees Celsius	5/9 (^{o}F-32)
Milligrams per liter (mg/l)	Parts per million (ppm)	Multiply by 1

APPENDIX E OXYGEN SOLUBILITY TABLE

Table A: Solubility of Oxygen in mg/l in Water Exposed to Water-Saturated Air at 760 mm Hg Pressure.
Salinity = Measure of quantity of dissolved salts in water.
Chlorinity = Measure of chloride content, by mass, of water.
$S(^0/_{00}) = 1.80655 \times \text{Chlorinity } (^0/_{00})$

Temp °C	**Chlorinity:0 Salinity:0**	**5.0 ppt 9.0 ppt**	**10.0 ppt 18.1 ppt**	**15.0 ppt 27.1 ppt**	**20.0 ppt 36.1 ppt**	**25.0 ppt 45.2 ppt**
0.0	14.62	13.73	12.89	12.10	11.36	10.66
1.0	14.22	13.36	12.55	11.78	11.07	10.39
2.0	13.83	13.00	12.22	11.48	10.79	10.14
3.0	13.46	12.66	11.91	11.20	10.53	9.90
4.0	13.11	12.34	11.61	10.92	10.27	9.66
5.0	12.77	12.02	11.32	10.66	10.03	9.44
6.0	12.45	11.73	11.05	10.40	9.80	9.23
7.0	12.14	11.44	10.78	10.16	9.58	9.02
8.0	11.84	11.17	10.53	9.93	9.36	8.83
9.0	11.56	10.91	10.29	9.71	9.16	8.64
10.0	11.29	10.66	10.06	9.49	8.96	8.45
11.0	11.03	10.42	9.84	9.29	8.77	8.28
12.0	10.78	10.18	9.62	9.09	8.59	8.11
13.0	10.54	9.96	9.42	8.90	8.41	7.95
14.0	10.31	9.75	9.22	8.72	8.24	7.79
15.0	10.08	9.54	9.03	8.54	8.08	7.64
16.0	9.87	9.34	8.84	8.37	7.92	7.50
17.0	9.67	9.15	8.67	8.21	7.77	7.36
18.0	9.47	8.97	8.50	8.05	7.62	7.22
19.0	9.28	8.79	8.33	7.90	7.48	7.09
20.0	9.09	8.62	8.17	7.75	7.35	6.96
21.0	8.92	8.46	8.02	7.61	7.21	6.84
22.0	8.74	8.30	7.87	7.47	7.09	6.72
23.0	8.58	8.14	7.73	7.34	6.96	6.61

Temp °C	Chlorinity:0 Salinity:0	5.0 ppt 9.0 ppt	10.0 ppt 18.1 ppt	15.0 ppt 27.1 ppt	20.0 ppt 36.1 ppt	25.0 ppt 45.2 ppt
24.0	8.42	7.99	7.59	7.21	6.84	6.50
25.0	8.26	7.85	7.46	7.08	6.72	6.39
26.0	8.11	7.71	7.33	6.96	6.62	6.28
27.0	7.97	7.58	7.20	6.85	6.51	6.18
28.0	7.83	7.44	7.08	6.73	6.40	6.09
29.0	7.69	7.32	6.96	6.62	6.30	5.99
30.0	7.56	7.19	6.85	6.51	6.20	5.90
31.0	7.43	7.07	6.73	6.41	6.10	5.81
32.0	7.31	6.96	6.62	6.31	6.01	5.72
33.0	7.18	6.84	6.52	6.21	5.91	5.63
34.0	7.07	6.73	6.42	6.11	5.82	5.55
35.0	6.95	6.62	6.31	6.02	5.73	5.46
36.0	6.84	3.52	6.22	5.93	5.65	5.38
37.0	6.73	6.42	6.12	5.84	5.56	5.31
38.0	6.62	6.32	6.03	5.75	5.48	5.23
39.0	6.52	6.22	5.98	5.66	5.40	5.15
40.0	6.41	6.12	5.84	5.58	5.32	5.08
41.0	6.31	6.03	5.75	5.49	5.24	5.01
42.0	6.21	5.93	5.67	5.41	5.17	4.93
43.0	6.12	5.84	5.58	5.33	5.09	4.86
44.0	6.02	5.75	5.50	5.25	5.02	4.79
45.0	5.93	5.67	5.41	5.17	4.94	4.72

* This table is provided for your information only. It is **NOT** required when calibrating the Model 85 in accordance with the instructions outlined in the section entitled Calibration.

APPENDIX F CALIBRATION VALUES TABLE

Table A: Calibration values for various atmospheric pressures and altitudes.
Note: This table is for your information only. It is not required for calibration.

Pressure Inches of Hg	Pressure mm Hg	Pressure kPA	Altitude in feet	Altitude in meters	Calibration Value in %
30.23	768	102.3	-276	-84	101
29.92	760	101.3	0	0	100
29.61	752	100.3	278	85	99
29.33	745	99.3	558	170	98
29.02	737	98.3	841	256	97
28.74	730	97.3	1126	343	96
28.43	722	96.3	1413	431	95
28.11	714	95.2	1703	519	94
27.83	707	94.2	1995	608	93
27.52	699	93.2	2290	698	92
27.24	692	92.2	2587	789	91
26.93	684	91.2	2887	880	90
26.61	676	90.2	3190	972	89
26.34	669	89.2	3496	1066	88
26.02	661	88.2	3804	1160	87
25.75	654	87.1	4115	1254	86
25.43	646	86.1	4430	1350	85
25.12	638	85.1	4747	1447	84
24.84	631	84.1	5067	1544	83
24.53	623	83.1	5391	1643	82
24.25	616	82.1	5717	1743	81
23.94	608	81.1	6047	1843	80
23.62	600	80.0	6381	1945	79
23.35	593	79.0	6717	2047	78
23.03	585	78.0	7058	2151	77
22.76	578	77.0	7401	2256	76
22.44	570	76.0	7749	2362	75
22.13	562	75.0	8100	2469	74
21.85	555	74.0	8455	2577	73
21.54	547	73.0	8815	2687	72
21.26	540	71.9	9178	2797	71
20.94	532	70.9	9545	2909	70
20.63	524	69.9	9917	3023	69
20.35	517	68.9	10293	3137	68

Y S I *i n c o r p o r a t e d*

1700/1725 Brannum Lane
Yellow Springs, Ohio 45387
+1 937 767-7241
800 765-4974 (US)
FAX (937) 767-9353
E-mail: environmental@YSI.com
Website: www.YSI.com

ITEM # 038503
DRW # A38503 - Web
Revision E - Web
November 1998

DR/890
COLORIMETER
PROCEDURES MANUAL

te/dk 03/13 10ed

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INTRODUCTION

This manual is divided into five sections:

Section 1 Chemical Analysis Information

This section applies to all the procedures. It provides background information and reference/review material for the technician or chemist. Commonly used techniques are explained in detail.

Section 2 Sample Pretreatment

This section provides a brief overview of sample pretreatment and two USEPA digestions. A brief discussion of the Hach Digesdahl Digestion Apparatus and the Hach Distillation Apparatus is included.

Section 3 Waste Management and Safety

Section 3 includes information on waste management, regulations, waste disposal and resources on waste management. The Safety portion covers reading an MSDS and general safety guidelines.

Section 4 Procedures

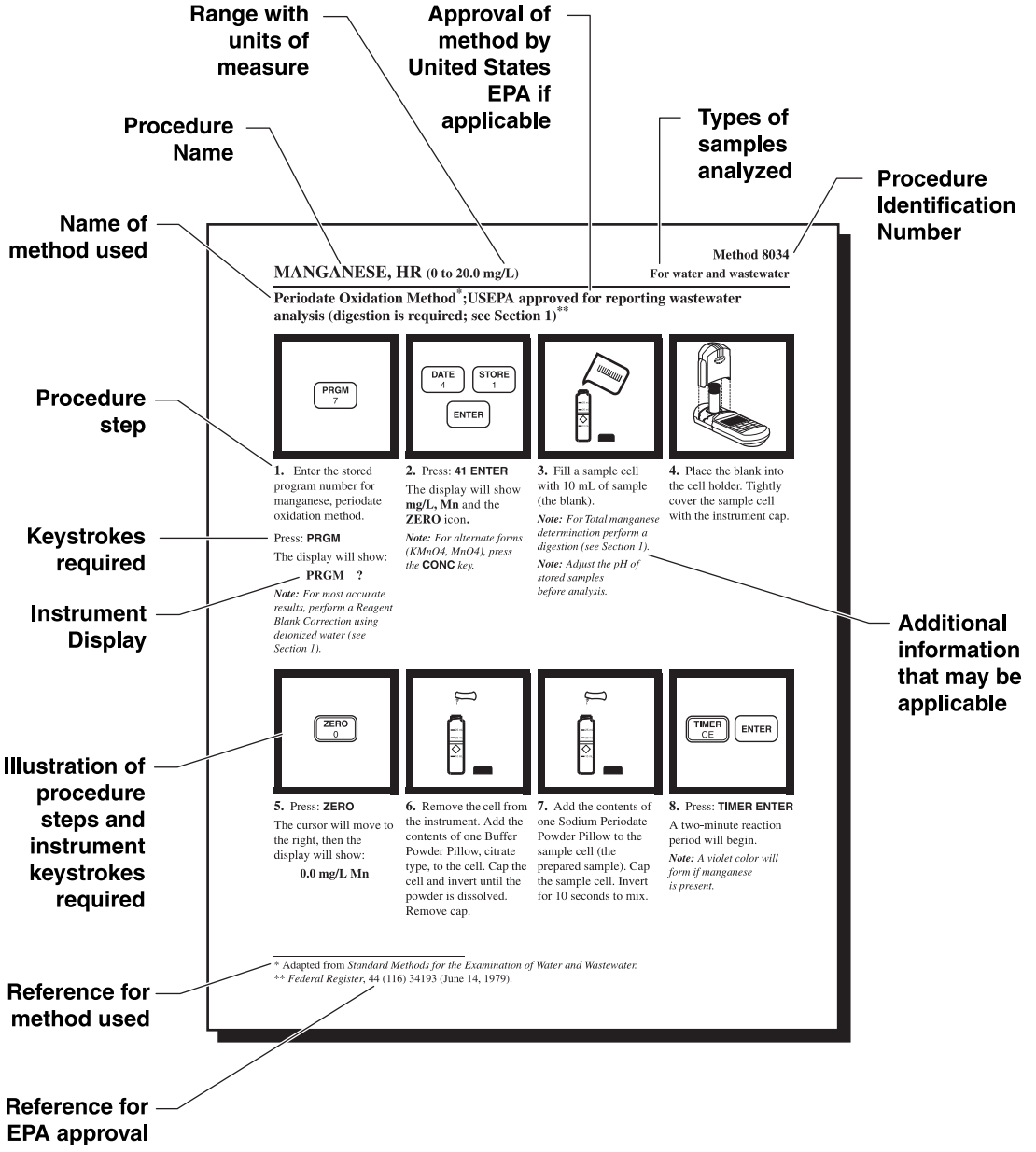
Section 4 contains step-by-step illustrated instructions for measuring parameters. The steps also include helpful notes. Each procedure contains information on sample collection, storage and preservation, accuracy checks, possible interferences, summary of method and a list of the reagents and apparatus necessary to run the test.

Section 5 Ordering Information

This section provides information needed for ordering, shipping, return of items and Hach trademarks.

Before attempting the analysis procedures the analyst should read the instrument manual to learn about the colorimeter's features and operation.

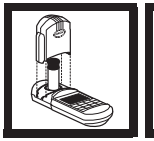
Sample Procedure Explained



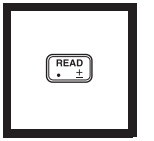
Specific sampling and storage information for this test

Confirm accuracy with these steps (in addition, may also be used to troubleshoot a test, improve technique, check reagents and to assure cleanliness of glassware)

MANGANESE, HR, continued



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L manganese will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-washed plastic bottles. Manganese may be lost by adsorption to glass container walls. Adjust the pH to less than 2 with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as manganese may be lost as a precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information. If only dissolved Mn is to be determined, filter before acid addition.

Accuracy Check

Standard Additions Method

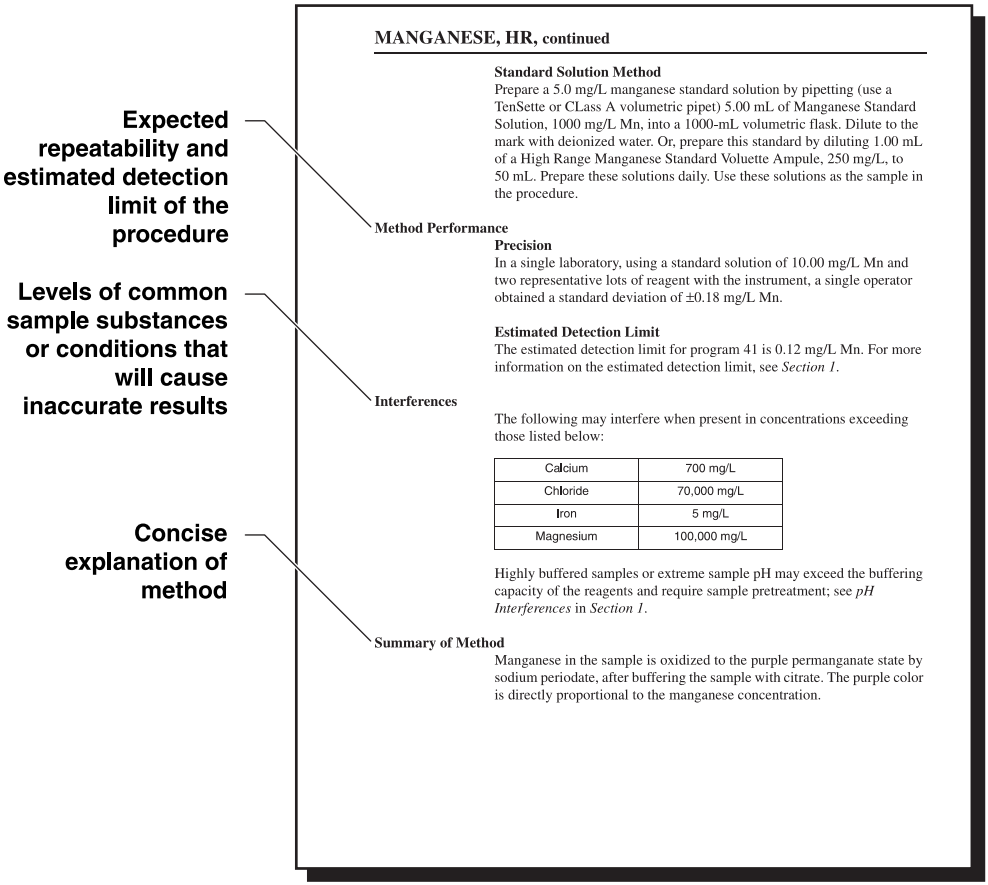
a) Snap the neck off a Manganese Voluette Ampule Standard Solution, 250 mg/L Mn.

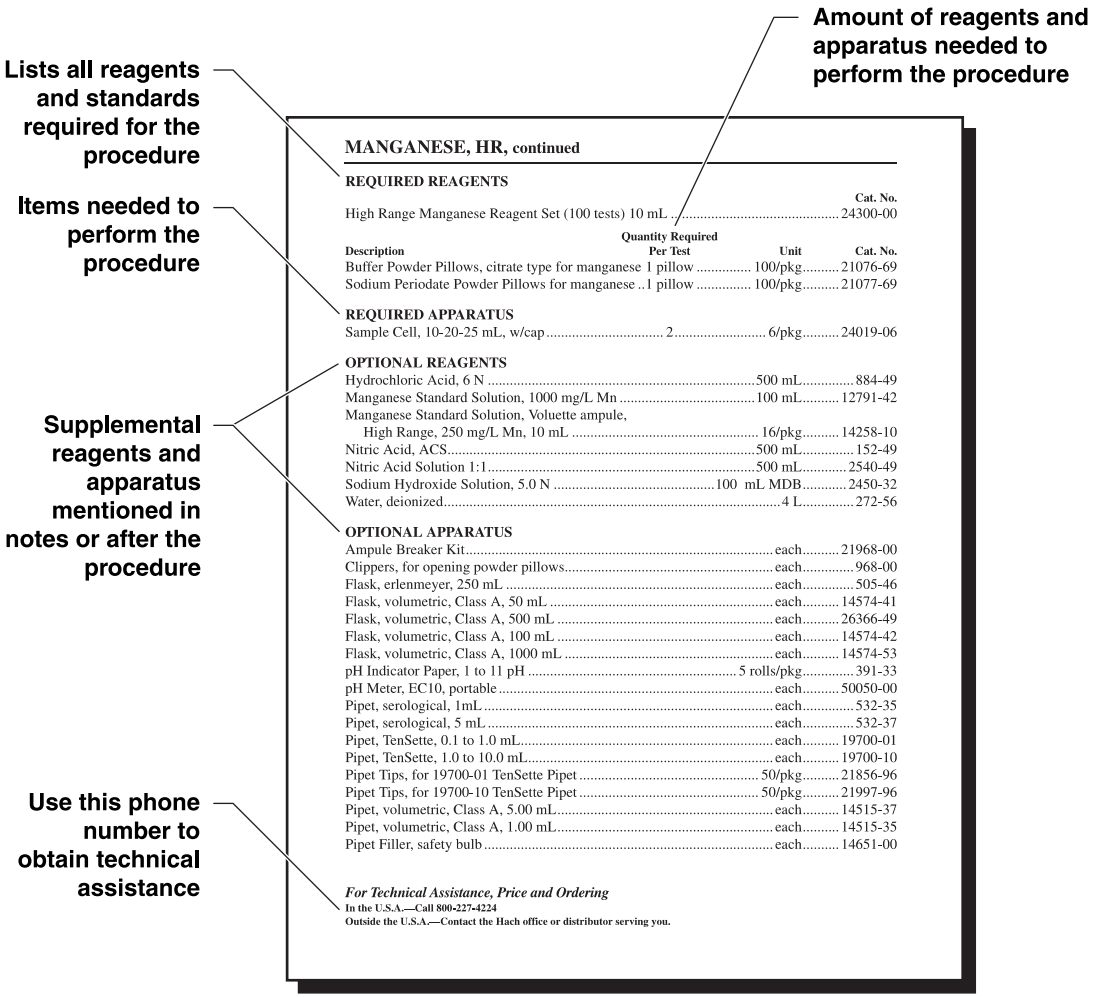
b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three 25-mL water samples. Swirl to mix.

c) Transfer only 10 mL of each solution to the 10-mL sample cells.

d) Analyze each standard addition sample as described in the procedure. The manganese concentration should increase 1.0 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.


Expected repeatability and estimated detection limit of the procedure
Levels of common sample substances or conditions that will cause inaccurate results
Concise explanation of method
MANGANESE, HR, continued
Standard Solution Method
Prepare a 5.0 mg/L manganese standard solution by pipetting (use a TenSette or CLass A volumetric pipet) 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Or, prepare this standard by diluting 1.00 mL of a High Range Manganese Standard Voluette Ampule, 250 mg/L, to 50 mL. Prepare these solutions daily. Use these solutions as the sample in the procedure.
Method Performance
Precision
In a single laboratory, using a standard solution of 10.00 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.18 mg/L Mn.
Estimated Detection Limit
The estimated detection limit for program 41 is 0.12 mg/L Mn. For more information on the estimated detection limit, see Section 1.
Interferences
The following may interfere when present in concentrations exceeding those listed below:
Calcium 700 mg/L
Chloride 70,000 mg/L
Iron 5 mg/L
Magnesium 100,000 mg/L
Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences in Section 1.
Summary of Method
Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese concentration.



MANGANESE, HR, continued

REQUIRED REAGENTS

			Cat. No.
High Range Manganese Reagent Set (100 tests) 10 mL			24300-00

Description	Quantity Required Per Test	Unit	Cat. No.
Buffer Powder Pillows, citrate type for manganese	1 pillow	100/pkg	21076-69
Sodium Periodate Powder Pillows for manganese	1 pillow	100/pkg	21077-69

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Hydrochloric Acid, 6 N	500 mL	884-49
Manganese Standard Solution, 1000 mg/L Mn	100 mL	12791-42
Manganese Standard Solution, Voluette ampule, High Range, 250 mg/L Mn, 10 mL	16/pkg	14258-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution 1:1	500 mL	2540-49
Sodium Hydroxide Solution, 5.0 N	100 mL MDB	2450-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Clippers, for opening powder pillows	each	968-00
Flask, erlenmeyer, 250 mL	each	505-46
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 500 mL	each	26366-49
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, serological, 1mL	each	532-35
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg	21997-96
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

SECTION 1 CHEMICAL ANALYSIS INFORMATION

Abbreviations

The following abbreviations are used throughout the text of the procedure section:

Abbrev-iation	Definition	Abbrev-iation	Definition
°C	degree(s) Celsius (Centigrade)	MDL	Method detection limit
°F	degree(s) Fahrenheit	MDB	marked dropping bottle
ACS	American Chemical Society reagent grade purity	mg/L	milligrams per liter (ppm)
APHA Standard Methods	*Standard Methods for the Examination of Water and Wastewater.*[1]	µg/L	micrograms per liter (ppb)
AV	AccuVac	mL	(milliliter)-approximately the same as a cubic centimeter (cc) or 1/1000 of a liter. Also known as a “cc”.
conc	concentrated	MR	medium range
CFR	Code of Federal Regulations	NIPDWR	National Interim Primary Drinking Water Regulations
DB	dropping bottle	NPDES	National Pollutant Discharge Elimination System
EDL	Estimated detection limit	PCB	Poly chlorinated biphenyl
FAU	Formazin Attenuation Units. Turbidity unit of measure based on a Formazin stock suspension.	SCDB	self-contained dropping bottle
g	grams	TNT	Test ‘N Tube™
gr/gal	grains per gallon (1 gr/gal = 17.12 mg/L)	TPH	Total petroleum hydrocarbons
HR	high range	TPTZ	(2,4,6-Tri-(2-Pyridyl)-1,3,5-Triazine)
L	Liter. Volume equal to one cubic decimeter (dm^3)	ULR	Ultra low range
LR	low range	USEPA	United States Environmental Protection Agency

[1] Published jointly by the American Public Health Association (APHA), the American Water Works Association (AWWA), and the Water Environment Federation (WEF). Order from Hach requesting Cat. No. 22708-00 or from the Publication Office of the American Public Health Association. This book is the standard reference work for water analysis. Many procedures contained in this manual are based on *Standard Methods.*

Converting Chemical Species

Species conversion factors for many commonly used substances are pre-programmed into the instrument (see *Table 1*). Conversions are method specific and are viewable after taking the reading by pressing **CONC**.

Table 1 Conversion Factors

To Convert From...	To...	Multiply By...
mg/L Al	mg/L Al_2O_3	1.8895
mg/L Ca-$CaCO_3$	mg/L Ca	0.4004
mg/L $CaCO_3$	mg/L Ca	0.4004
mg/L $CaCO_3$	mg/L Mg	0.2428
µg/L Carbohydrazide	µg/L Hydroquinone	1.92
µg/L Carbohydrazide	µg/L ISA	2.69
µg/L Carbohydrazide	µg/L MEKO	3.15
mg/L Cr^{6+}	mg/L CrO_4^{2-}	2.231
mg/L Cr^{6+}	mg/L Na_2CrO_4	3.115
mg/L Mg-$CaCO_3$	mg/L Mg	0.2428
mg/L Mn	mg/L $KMnO_4$	2.876
mg/L Mn	mg/L MnO_4^-	2.165
mg/L Mo^{6+}	mg/L MoO_4^{2-}	1.667
mg/L Mo^{6+}	mg/L Na_2MoO_4	2.146
mg/L N	mg/L NH_3	1.216
mg/L N	mg/L NO_3^-	4.427
mg/L Na_2CrO_4	mg/L Cr^{6+}	0.321
mg/L Na_2CrO_4	mg/L CrO_4^{2-}	0.72
mg/L NH_2Cl-N	mg/L Cl_2	5.0623
mg/L NH_2Cl-N	mg/L NH_2Cl	3.6750
mg/L NH_3-N	mg/L NH_3	1.216
mg/L NH_3-N	mg/L NH_4^+	1.288
mg/L NO_2^-	mg/L $NaNO_2$	1.5
mg/L NO_2^-	mg/L NO_2^--N	0.3045
mg/L NO_2^--N	mg/L $NaNO_2$	4.926
µg/L NO_2^--N	µg/L $NaNO_2$	4.926
mg/L NO_2^--N	mg/L NO_2^-	3.284
µg/L NO_2^--N	µg/L NO_2^-	3.284
mg/L NO_3^--N	mg/L NO_3^-	4.427
mg/L PO_4^{3-}	mg/L P	0.3261
µg/L PO_4^{3-}	µg/L P	0.3261
mg/L PO_4^{3-}	mg/L P_2O_5	0.7473
µg/L PO_4^{3-}	µg/L P_2O_5	0.7473
mg/L SiO_2	mg/L Si	0.4674
µg/L SiO_2	µg/L Si	0.4674

Hardness Conversion

Table 2 lists the factors for converting one unit of measure for hardness to another unit of measure. For example, to convert mg/L $CaCO_3$ to German parts/100,000 CaO, multiply the value in mg/L x 0.056.

Table 2 Hardness Conversion Factors

Units of Measure	mg/L $CaCO_3$	British gr/gal (Imperial) $CaCO_3$	American gr/gal (US) $CaCO_3$	French parts/ 100,000 $CaCO_3$	German Parts/ 100,000 CaO	meq/L[1]	g/L CaO	lbs./cu ft $CaCO_3$
mg/L $CaCO_3$	1.0	0.07	0.058	0.1	0.056	0.02	$5.6x10^{-4}$	$6.23x10^{-5}$
English gr/gal $CaCO_3$	14.3	1.0	0.83	1.43	0.83	0.286	$8.0x10^{-3}$	$8.9x10^{-4}$
US gr/gal $CaCO_3$	17.1	1.2	1.0	1.72	0.96	0.343	$9.66x10^{-3}$	$1.07x10^{-3}$
Fr. p/ 100,000 $CaCO_3$	10.0	0.7	0.58	1.0	0.56	0.2	$5.6x10^{-3}$	$6.23x10^{-4}$
Ger. p/ 100,000 CaO	17.9	1.25	1.04	1.79	1.0	0.358	$1x10^{-2}$	$1.12x10^{-3}$
meq/L	50.0	3.5	2.9	5.0	2.8	1.0	$2.8x10^{-2}$	$3.11x10^{-2}$
g/L CaO	1790.0	125.0	104.2	179.0	100.0	35.8	1.0	0.112
lbs./cu ft $CaCO_3$	16,100.0	1,123.0	935.0	1,610.0	900.0	321.0	9.0	1.0

[1] 'epm/L, or 'mval/L'
Note: 1 meq/L = 1N/1000

Dissolved Oxygen

Table 3 lists the mg/L dissolved oxygen in water at saturation for various temperatures and atmospheric pressures. The table was formulated in a laboratory using pure water. The values given are only approximations for estimating the oxygen content of a particular body of surface water.

Table 3 Dissolved Oxygen Saturation In Water

		Pressure in Millimeters and Inches Hg							
		mm							
		775	760	750	725	700	675	650	625
Temp		inches							
°F	°C	30.51	29.92	29.53	28.45	27.56	26.57	25.59	24.61
32.0	0	14.9	14.6	14.4	13.9	13.5	12.9	12.5	12.0
33.8	1	14.5	14.2	14.1	13.6	13.1	12.6	12.2	11.7
35.6	2	14.1	13.9	13.7	13.2	12.9	12.3	11.8	11.4
37.4	3	13.8	13.5	13.3	12.9	12.4	12.0	11.5	11.1
39.2	4	13.4	13.2	13.0	12.5	12.1	11.7	11.2	10.8
41.0	5	13.1	12.8	12.6	12.2	11.8	11.4	10.9	10.5
42.8	6	12.7	12.5	12.3	11.9	11.5	11.1	10.7	10.3
44.6	7	12.4	12.2	12.0	11.6	11.2	10.8	10.4	10.0
46.4	8	12.1	11.9	11.7	11.3	10.9	10.5	10.1	9.8
48.2	9	11.8	11.6	11.5	11.1	10.7	10.3	9.9	9.5
50.0	10	11.6	11.3	11.2	10.8	10.4	10.1	9.7	9.3
51.8	11	11.3	11.1	10.9	10.6	10.2	9.8	9.5	9.1
53.6	12	11.1	10.8	10.7	10.3	10.0	9.6	9.2	8.9
55.4	13	10.8	10.6	10.5	10.1	9.8	9.4	9.1	8.7
57.2	14	10.6	10.4	10.2	9.9	9.5	9.2	8.9	8.5
59.0	15	10.4	10.2	10.0	9.7	9.3	9.0	8.7	8.3
60.8	16	10.1	9.9	9.8	9.5	9.1	8.8	8.5	8.1
62.6	17	9.9	9.7	9.6	9.3	9.0	8.6	8.3	8.0
64.4	18	9.7	9.5	9.4	9.1	8.8	8.4	8.1	7.8
66.2	19	9.5	9.3	9.2	8.9	8.6	8.3	8.0	7.6
68.0	20	9.3	9.2	9.1	8.7	8.4	8.1	7.8	7.5
69.8	21	9.2	9.0	8.9	8.6	8.3	8.0	7.7	7.4
71.6	22	9.0	8.8	8.7	8.4	8.1	7.8	7.5	7.2
73.4	23	8.8	8.7	8.5	8.2	8.0	7.7	7.4	7.1

Table 3 Dissolved Oxygen Saturation In Water (continued)

		Pressure in Millimeters and Inches Hg							
		mm							
		775	760	750	725	700	675	650	625
Temp		inches							
°F	°C	30.51	29.92	29.53	28.45	27.56	26.57	25.59	24.61
75.2	24	8.7	8.5	8.4	8.1	7.8	7.5	7.2	7.0
77.0	25	8.5	8.4	8.3	8.0	7.7	7.4	7.1	6.8
78.8	26	8.4	8.2	8.1	7.8	7.6	7.3	7.0	6.7
80.6	27	8.2	8.1	8.0	7.7	7.4	7.1	6.9	6.6
82.4	28	8.1	7.9	7.8	7.6	7.3	7.0	6.7	6.5
84.2	29	7.9	7.8	7.7	7.4	7.2	6.9	6.6	6.4
86.0	30	7.8	7.7	7.6	7.3	7.0	6.8	6.5	6.2
87.8	31	7.7	7.5	7.4	7.2	6.9	6.7	6.4	6.1
89.6	32	7.6	7.4	7.3	7.0	6.8	6.6	6.3	6.0
91.4	33	7.4	7.3	7.2	6.9	6.7	6.4	6.2	5.9
93.2	34	7.3	7.2	7.1	6.8	6.6	6.3	6.1	5.8
95.0	35	7.2	7.1	7.0	6.7	6.5	6.2	6.0	5.7
96.8	36	7.1	7.0	6.9	6.6	6.4	6.1	5.9	5.6
98.6	37	7.0	6.8	6.7	6.5	6.3	6.0	5.8	5.6
100.4	38	6.9	6.7	6.6	6.4	6.2	5.9	5.7	5.5
102.2	39	6.8	6.6	6.5	6.3	6.1	5.8	5.6	5.4
104.0	40	6.7	6.5	6.4	6.2	6.0	5.7	5.5	5.3
105.8	41	6.6	6.4	6.3	6.1	5.9	5.6	5.4	5.2
107.6	42	6.5	6.3	6.2	6.0	5.8	5.6	5.3	5.1
109.4	43	6.4	6.2	6.1	5.9	5.7	5.5	5.2	5.0
111.2	44	6.3	6.1	6.0	5.8	5.6	5.4	5.2	4.9
113.0	45	6.2	6.0	5.9	5.7	5.5	5.3	5.1	4.8
114.8	46	6.1	5.9	5.9	5.6	5.4	5.2	5.4	4.8
116.6	47	6.0	5.9	5.8	5.6	5.3	5.1	4.8	4.7
118.4	48	5.9	5.8	5.7	5.5	5.3	5.0	4.8	4.6
120.2	49	5.8	5.7	5.6	5.4	5.2	5.0	4.7	4.5
122.0	50	5.7	5.6	5.5	5.3	5.1	4.9	4.7	4.4

Sample Collection, Preservation and Storage

Correct sampling and storage are critical for accurate testing. For greatest accuracy, thoroughly clean sampling devices and containers to prevent carryover from previous samples. Preserve the sample properly; each procedure has information about sample preservation.

- The least expensive containers are polypropylene or polyethylene.
- The best and most expensive containers are quartz or PTFE (polytetrafluoroethylene, Teflon).
- Avoid soft glass containers for metals in the microgram-per-liter range.
- Store samples for silver determination in light-absorbing containers, such as amber bottles.

Avoid contaminating the sample with metals from containers, deionized water or membrane filters. Thoroughly clean sample containers as described under Acid Washing Bottles.

Preservation slows the chemical and biological changes that continue after collection. These processes may change the amount of a chemical species available for analysis. Normally, analyze the samples as soon as possible after collection, especially when the analyte concentration is expected to be low. This also reduces the chance for error and minimizes labor.

Preservation methods include pH control, chemical addition, refrigeration and freezing. *Table 4* gives the recommended preservation for various substances. It also includes suggested types of containers and the maximum recommended holding times for properly preserved samples.

Preserve aluminum, cadmium, chromium, cobalt, copper, iron, lead, nickel, potassium, silver and zinc samples for at least 24 hours by adding one Nitric Acid Solution Pillow 1:1 (Cat. No. 2540-98) per liter of sample. Check the pH with pH indicator paper or a pH meter to assure the pH is 2 or less. Add additional pillows if necessary. Adjust the sample pH prior to analysis by adding an equal number of Sodium Carbonate Anhydrous Powder Pillows (Cat. No. 179-98). Or raise the pH to 4.5 with Sodium Hydroxide Standard Solution, 1 N or 5 N. Correct for the added volume of the preservatives; see *Correcting For Volume Additions.*

Table 4 Required Containers, Preservation Techniques and Holding Times[1]

Parameter No./Name	Container[2]	Preservation[3,4]	Maximum Holding Time[5]
Table 1A - Bacterial Tests:			
1-4. Coliform, fecal and total	P,G	Cool, 4°C, 0.008%, $Na_2S_2O_3$[6]	6 hours
5. Fecal streptococci	P,G	Cool, 4°C, 0.008%, $Na_2S_2O_3$	6 hours
Table 1B - Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days
2. Alkalinity	P, G	Cool, 4°C	14 days
4. Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days
9. Biochemical oxygen demand (BOD)	P, G	Cool, 4°C	48 hours
11. Bromide	P, G	None required	28 days
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours
15. Chemical oxygen demand	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days
16. Chloride	P, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze immediately
21. Color	P, G	Cool, 4°C	48 hours
23-24. Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH>12, 0.6 g ascorbic acid[6]	14 days[7]
25. Fluoride	P	None required	28 days
27. Hardness	P, G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately
31, 43. Kjeldahl and organic nitrogen	P, G	Cool 4°C, H_2SO_4 to pH<2	28 days
Metals:[8]			
18. Chromium VI	P, G	Cool, 4°C	24 hours
35. Mercury	P, G	HNO_3 to pH<2	6 months
3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75.[9] Metals, except boron, chromium VI and mercury	P, G	do	6 months
38. Nitrate	P, G	Cool, 4°C	48 hours
39. Nitrate-nitrite	P, G	Cool 4°C, H_2SO_4 to pH<2	28 days
40. Nitrite	P, G	Cool, 4°C	48 hours
41. Oil and grease	G	Cool, 4°C, HCl or H_2SO_4 to pH<2	28 days
42. Organic Carbon	P, G	Cool, 4°C, HCl or H_2SO4 or H_3PO_4 to pH<2	28 days
44. Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
46. Oxygen, dissolved probe	G Bottle and top	None required	Analyze immediately
47. Winkler	G Bottle and top	Fix on site and store in dark	8 hours

Table 4 Required Containers, Preservation Techniques and Holding Times[1] (continued)

Parameter No./Name	Container[2]	Preservation[3,4]	Maximum Holding Time[5]
48. Phenols	G only	Cool 4°C, H_2SO_4 to pH<2	28 days
49. Phosphorus, elemental	G	Cool, 4°C	48 hours
50. Phosphorus, total	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days
53. Residue, total	P, G	Cool, 4°C	7 days
54. Residue, filterable	P, G	Cool, 4°C	7 days
55. Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
56. Residue, Settleable	P, G	Cool, 4°C	48 hours
57. Residue, volatile	P, G	Cool, 4°C	7 days
61. Silica	P, PFTE or quartz	Cool, 4°C	28 days
64. Specific conductance	P, G	Cool, 4°C	28 days
65. Sulfate	P, G	Cool, 4°C	28 days
66. Sulfide	P, G	Cool 4°C, add zinc acetate plus sodium hydroxide to pH>9	7 days
67. Sulfite	P, G	none required	Analyze immediately
68. Surfactants	P, G	Cool, 4°C	48 hours
69. Temperature	P, G	None required	Analyze immediately
73. Turbidity	P, G	Cool, 4°C	48 hours

1 This table was taken from Table II published in the Federal Register, July 1, 1995, 40 CFR, Part 136.3, pages 643-645. Organic tests are not included.

2 Polyethylene (P) or glass (G).

3 Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

4 When any sample is to be shipped by common carrier or sent through United States Mails, it must comply with the Department of Transportation Hazardous Material Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

5 Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permitee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administer under §136.3(e). Some samples may not be stable for the maximum time period given in the table. A permitee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See §136.3(e) for details. The term “analyze immediately” usually means within 15 minutes or less after sample collection.

6 Should only be used in the presence of residual chlorine.

7 Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

8 Samples should be filtered immediately on-site before adding preservative for dissolved metals.

9 Numbers refer to parameter numbers in 40 CFR Part 136.3, Table 1B.

Collecting Water Samples

Obtain the best sample by careful collection. In general, collect samples near the center of the vessel or duct and below the surface. Use only clean containers (bottles, beakers). Rinse the container several times first with the water to be sampled.

Take samples as close as possible to the source of the supply. This lessens the influences of the distribution system on the sample. Let the water run long enough to flush the system. Fill sample containers slowly with a gentle stream to avoid turbulence and air bubbles. Collect water samples from wells after the pump has run long enough to deliver water representative of the ground water feeding the well.

It is hard to obtain a truly representative sample when collecting surface water samples. Obtain best results by testing several samples. Use samples taken at different times from several locations and depths. The results can be used to establish patterns for that particular body of water.

Generally, as little time as possible should elapse between collecting the sample and analyzing it.

Depending on the test, special precautions in handling the sample may be necessary. This prevents natural interferences such as organic growth or loss or gain of dissolved gases. Each procedure describes sample preservatives and storage techniques for samples that are held for testing.

Acid Washing Bottles

If a procedure suggests acid-washing, use the following instructions:

a) Clean the glassware or plasticware with laboratory detergent (phosphate-free detergent is recommended).

b) Rinse well with tap water.

c) Rinse with a 1:1 Hydrochloric Acid Solution or 1:1 Nitric Acid Solution.

d) Rinse well with deionized water at least four times. Up to 12-15 rinses may be necessary if chromium is being determined.

e) Air dry.

Use chromic acid or chromium-free substitutes to remove organic deposits from glass containers. Rinse containers thoroughly with water to remove traces of chromium.

Wash glassware for phosphate determinations with phosphate-free detergents and acid-wash with 1:1 HCl. Thoroughly rinse the glassware with deionized water. For ammonia and Kjeldahl nitrogen, rinse with ammonia-free water.

Correcting for Volume Additions

If you use a large volume of preservative, correct for the volume of preservative added. This accounts for dilution due to the acid added to preserve the sample and the base used to adjust the pH to the range of the procedure. This correction is made as follows:

1. Determine the volume of initial sample, the volume of acid and base added, and the total or final volume of the sample.

2. Divide the total volume by the initial volume of sample.

3. Multiply the test result by this factor.

Example:

A one-liter sample was preserved with 2 mL of nitric acid. It was neutralized with 5 mL of 5 N sodium hydroxide. The result of the analysis procedure was 10.00 mg/L. What is the volume correction factor and correct result?

1. Total Volume = 1000 mL + 2 mL + 5 mL = 1007 mL

2. $\frac{1007}{1000}$ = 1.007 = volume correction factor

3. 10.0 mg/L × 1.007 = 10.07 mg/L = correct result

Hach 1:1 Nitric Acid Pillows contain 2.5 mL of acid; correct for this volume. The addition of a Sodium Carbonate Power Pillow (neutralizes the 1:1 Nitric Acid Solution Pillow) does not need to be corrected for.

Boiling Aids

Boiling is necessary in some procedures. Using a boiling aid such as boiling chips (Cat. No. 14835-31) helps reduce bumping. Bumping is caused by the sudden, almost explosive conversion of water to steam as it is heated. Avoid bumping; it may cause injury or sample loss.

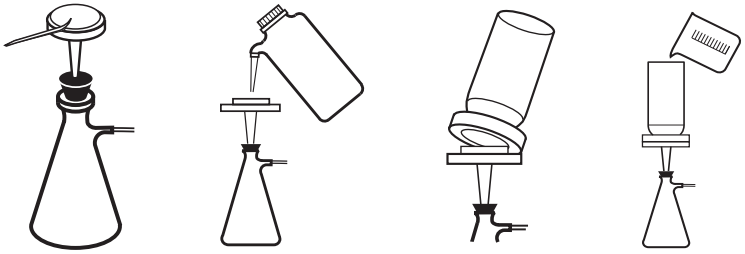
Make sure the boiling aids will not contaminate the sample. Do not use boiling aids (except glass beads) more than once. Loosely covering the sample during boiling will prevent splashing, reduce the chances of contamination and minimize sample loss.

Sample Filtration

Filtering separates particles from the aqueous sample. Filtration uses a medium, usually filter paper, to retain particles but pass solution. This is especially helpful when sample turbidity interferes with analysis. Two general methods of filtration are gravity and vacuum. Gravity filtration uses gravity to pull the sample though the filter paper. Vacuum filtration uses suction and gravity to move the sample through the filter. An aspirator or vacuum pump creates the suction. Vacuum filtration is faster than gravity filtration. Vacuum filter (see *Figure 1*) as follows:

1. Using tweezers, place a filter paper into the filter holder.
2. Place the filter holder assembly in the filtering flask. Wet the filter with deionized water to ensure adhesion to the holder. Empty the flask before filtering the sample.
3. Position the funnel housing on the filter holder assembly.
4. While applying a vacuum to the filtering flask, transfer the sample to the filtering apparatus.
5. Slowly release the vacuum from the filtering flask and transfer the solution from the filter flask to another container.

Figure 1 Vacuum Filtration



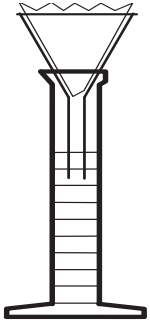
REQUIRED APPARATUS FOR VACUUM FILTRATION

Description	Unit	Cat. No.
Filter Discs, glass 47 mm, 1.5 µm	100/pkg	2530-00
Filter Holder, membrane	each	13529-00
Flask, filter, 500 mL	each	546-49
Pump, vacuum, hand operated	each	14283-00
OR		
Pump, vacuum, portable, 115 V	each	14697-00
Pump, vacuum, portable, 230 V	each	14697-02

Several procedures in this manual use gravity filtration. The only labware required is filter paper, a conical funnel and a receiving vessel. This labware is included under Optional Apparatus at the end of a procedure. Gravity filtration is better for retaining fine particles. For faster filtering, add solution until the filter paper cone is three-fourths filled. Never fill the cone completely. Gravity filter (see *Figure 2*) as follows:

1. Place a filter paper into the funnel.
2. Wet the filter with deionized water to ensure adhesion to the funnel. Allow all the deionized water to drain.
3. Place the funnel into an erlenmeyer flask or graduated cylinder.
4. Pour the sample into the funnel.

Figure 2 Gravity Filtration



REQUIRED APPARATUS FOR GRAVITY FILTRATION

Description	Unit	Cat No.
Cylinder, graduated, 100 mL	each	508-42
Funnel, poly, 65 mm	each	1083-67
Filter Paper, 12.5 cm	each	1894-57
Flask, erlenmeyer, 125 mL	each	505-43

Testing for metals requires acid and heat to pretreat the sample. Since these conditions destroy filter paper, vacuum filtration with glass fiber filter discs is recommended. Also, glass filter discs, unlike paper, do not retain colored species.

Temperature Considerations

For best results, perform most tests in this manual with sample temperatures between 20 °C (68 °F) and 25 °C (77 °F). If a test requires closer temperature control, notes in the procedure will indicate this.

Sample Dilution Techniques

Ten and 25 mL are the volumes used for most colorimetric tests. However, in some tests, the color developed in the sample may be too intense to be measured. Unexpected colors may develop in other tests. In both cases, dilute the sample to determine if interfering substances are present.

To dilute the sample easily, pipet the chosen sample portion into a clean graduated cylinder (or volumetric flask for more accurate work). Fill the cylinder (or flask) to the desired volume with deionized water. Mix well. Use the diluted sample when running the test.

To help with dilutions, *Table 5* shows the amount of sample used, the amount of deionized water used to bring the volume up to 25 mL and the multiplication factor.

The concentration of the sample is equal to the diluted sample reading multiplied by the multiplication factor.

More accurate dilutions can be done with a pipet and a 100-mL volumetric flask (see *Table 6* for more information). Pipet the sample and dilute to volume with deionized water. Swirl to mix.

Table 5 Sample Dilution Volumes

Sample Volume (mL)	mL Deionized Water Used to Bring the Volume to 25 mL	Multiplication Factor
25.0	0.0	1
12.5	12.5	2
10.0[1]	15.0	2.5
5.0[1]	20.0	5
2.5[1]	22.5	10
1.0[1]	24.0	25
0.250[1]	24.75	100

[1] For sample sizes of 10 mL or less, use a pipet to measure the sample into the graduated cylinder or volumetric flask.

Table 6 Multiplication Factors for Diluting to 100 mL

Sample Volume (mL)	Multiplication Factor
1	100
2	50
5	20
10	10
25	4
50	2

Sample Dilution and Interfering Substances

Sample dilution may influence the level at which a substance may interfere. The effect of the interferences decreases as the dilution increases. In other words, higher levels of an interfering substance can be present in the original sample if it is diluted before analysis.

An Example:

Copper does not interfere at or below 100 mg/L for a 25.00 mL sample in a procedure. If the sample volume is diluted with an equal volume of water, what is the level at which copper will not interfere?

$$\frac{\text{Total volume}}{\text{Sample volume}} = \text{Dilution factor}$$

$$\frac{25}{12.5} = 2$$

$$\text{Interference Level} \times \text{Dilution Factor} = \text{Interference level in sample}$$

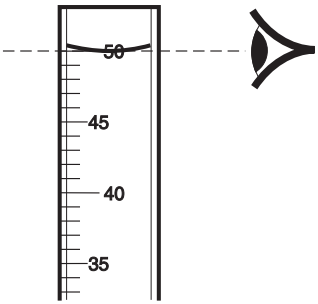
$$100 \times 2 = 200$$

The level at which copper will not interfere in the undiluted sample is at or below 200 mg/L.

Using Pipets and Graduated Cylinders

When small sample quantities are used, the accuracy of measurements is important. *Figure 3* illustrates the proper way of reading the sample level or the meniscus formed when the liquid wets the cylinder or pipet walls.

Figure 3 Reading the Meniscus



Rinse the pipet or cylinder two or three times with the sample to be tested before filling. Use a pipet filler or pipet bulb to draw the sample into the pipet. Never pipet chemical reagent solutions or samples by mouth. When filling a pipet, keep the tip of the pipet below the surface of the sample as the sample is drawn into the pipet.

Serological pipets have marks that indicate the volume of liquid delivered by the pipet. The marks may extend to the tip of the pipet or may be only on the straight portion of the tube. If the marks are only on the straight part of the tube, fill serological pipets to the zero mark and discharge the sample by draining the sample until the meniscus is level with the desired mark. If the serological pipet has marks extended to the tip of the pipet, fill the pipet to the desired volume and drain all the sample from the pipet. Then blow the sample out of the pipet tip for accurate measurements.

Volumetric (transfer) pipets have a bulb in the middle and a single ring above the bulb to indicate the volume of liquid when it is filled to the mark. To discharge a volumetric pipet, hold the pipet vertical until only a small amount of liquid remains (about ¾ inch), then hold the pipet at a slight angle against the container wall to drain. Do not attempt to discharge the solution remaining in the tip of the pipet after draining. Volumetric pipets are designed to retain a small amount of sample in the pipet tip.

If sample drops stay on the walls of the pipet, the pipet is dirty and is not delivering the correct amount of sample. Wash the pipet thoroughly with a laboratory detergent or cleaning solution and rinse several times with deionized water.

Using the TenSette Pipet

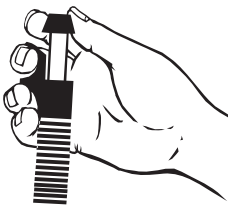
For best results use a new tip each time you pipet. After several uses, the pipet tip may retain some liquid, causing inaccurate delivery. Each pipet is supplied with 50 tips; order Hach replacement tips for best results.

Always use careful, even hand movements for best reproducibility. If the pipet does not operate smoothly, disassemble and coat the piston and retainer with high-quality stopcock grease. Also coat the metering turret lightly with grease. Refer to the TenSette Pipet manual.

For best pipetting accuracy, the solution and the room temperature should be between 20-25 °C.

Never lay the pipet down with the liquid in the tip. Solution could leak into the pipet and cause corrosion.

Operating the TenSette Pipet



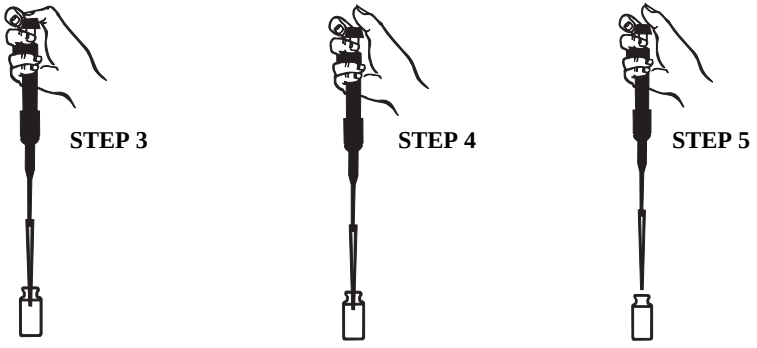
1. Attach a clean tip by holding the pipet body in one hand and gently pressing the large end of the pipet tip onto the tapered end of the pipet. Be sure a good seal is obtained.

2. Turn the turret cap to align the desired volume with the mark on the pipet body.

3. Using a smooth motion, press down on the turret cap until it reaches the stop. Immerse the tip about 5 mm (¼ inch) below the solution surface to avoid drawing air into the pipet. Do not insert the tip any deeper or the delivery volume may be affected.

4. While maintaining a constant pressure, allow the turret to return slowly to the extended position. A rapid return may affect the delivery volume.

5. With the turret up, take the tip out of the solution and move it to the receiving vessel. Do not press on the turret cap while moving the pipet.




STEP 6

6. Use the thumb and forefinger to twist the turret cap to the next higher volume position to ensure quantitative transfer of the sample. The “F” position provides full blowout.


STEP 7

7. With the tip in contact with the side of the receiving vessel, slowly and smoothly press down on the turret cap until it reaches the stop and the solution is completely discharged.

Mixing Water Samples

The following two methods may be helpful in tests that require mixing sample with chemicals (usually indicated by "invert to mix" instructions).

1. When mixing sample in a round sample cell or mixing cylinder, invert the cell or cylinder; see *Figure 4*. Hold the cell in a vertical position with the cap on top. Invert the cell so the cap is on the bottom. Return the cell to the original position. Do the same with the mixing cylinder.

2. Swirling is recommended when mixing samples in a graduated cylinder or a titration flask. Grip the cylinder (or flask) firmly with the tips of three fingers; see *Figure 5*. Hold the cylinder at a 45-degree angle and twist the wrist. This should move the cylinder in an approximately 12-inch circle, creating enough rotation to complete the mixing in a few turns.

These mixing procedures are the most gentle. Both methods are simple but take a bit of practice to obtain the best results.

Figure 4 Inverting a Sample Cell

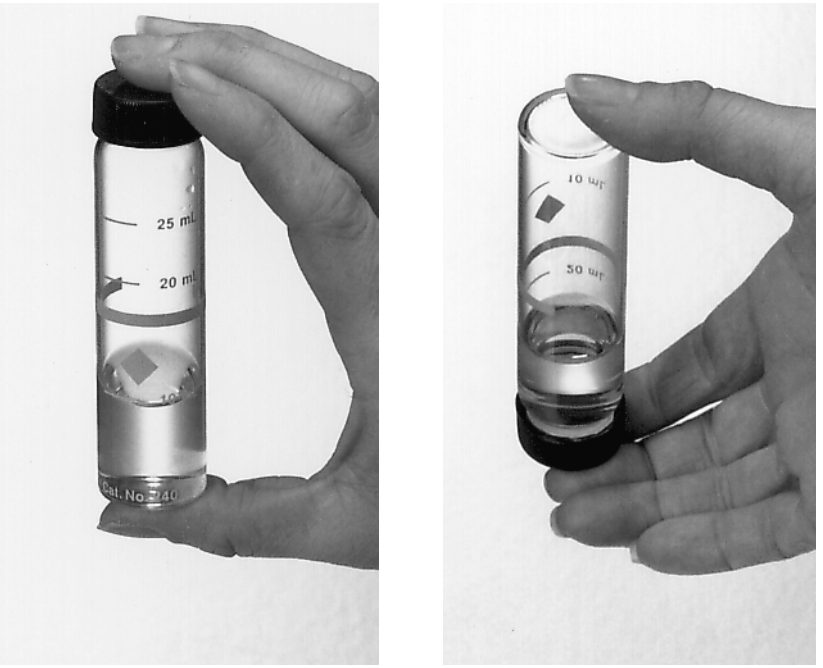
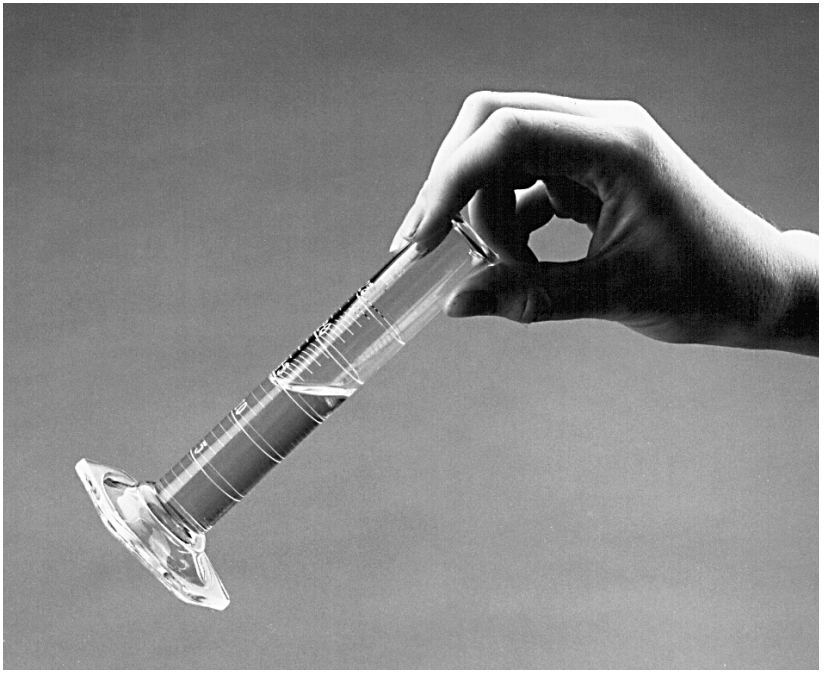


Figure 5 Swirling a Graduated Cylinder



Using Sample Cells

Orientation of Sample Cells

Two round sample cells are shipped with the DR/820, DR/850 and DR/890. They are marked with 10-, 20- and 25-mL fill lines which may be used to measure the sample volume unless the procedure instructs you to use other glassware to measure the sample volume.

To minimize variability of measurements using a particular cell, always place the cell into the cell holder with the same orientation. The cells are placed in the instrument with the fill marks facing the user.

In addition to proper orientation, the sides of the cells should be free of smudges, fingerprints, etc. to ensure accurate readings. Wipe the sides of the cells with a moist cloth followed by a dry soft cloth to clean the surface before taking measurements.

Care of Hach Sample Cells

Store sample cells in their boxes when not in use to protect them from scratching and breaking. It is good laboratory practice to empty and clean sample cells after analyses are complete--avoid leaving colored solutions in the cells for extended periods of time. Finish the cleaning procedure with a few rinses of deionized water and allow to dry. Individual procedures often recommend specific cleaning methods.

Cleaning Sample Cells

Most laboratory detergents can be used at recommended concentrations. Neutral detergents such as Neutracon are safer if regular cleaning is required, as in the case of protein residues.

If using a detergent, you can speed cleaning by increasing the temperature or using an ultrasonic bath.

Rinsing is more efficient when using deionized water.

Using the COD/TNT Adapter

Use care when seating a vial into the COD/ TNT adapter (for COD vials and Test ‘N Tubes). Place the vial into the adapter and press straight down on the top of the vial until it seats solidly. Do not move the vial from side to side; this can cause errors.

Volume Measurement Accuracy

The sample cells supplied with the instrument have fill marks to indicate 10, 20 or 25 mL. The fill marks are intended to measure the volume to be analyzed. Do not use these fill marks to perform sample dilutions.

If a sample must be diluted, use a pipet, graduated mixing cylinder and/or a volumetric flask for accurate measurement. When diluting, accuracy is important because a slight mistake in measuring a small sample will cause

a substantial error in the result. For instance, a 0.1-mL mistake in the dilution of a 1.0-mL final volume produces a 10% error in the test result.

Volumes for standard additions can be measured using the 25-mL mark, but it is not recommended for the 10-mL mark due to a potentially excessive relative error. An error of 0.5 mL in 25 mL is only 2%, while 0.5 mL error in 10 mL is 5%.

For 10 mL standard additions, follow this procedure:

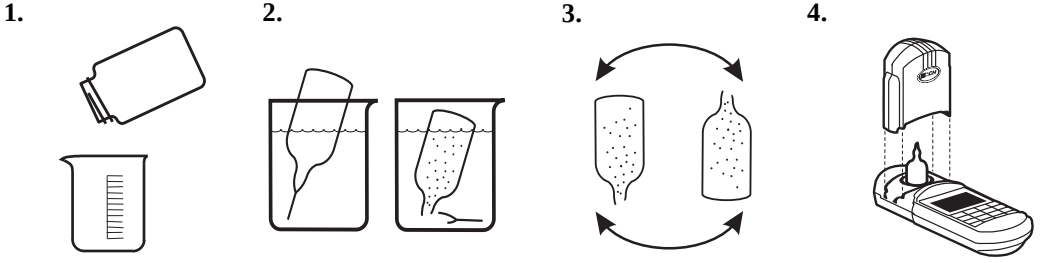
1. Transfer 10.0 mL of sample into a clean, dry sample cell (the unspiked sample).
2. Add the standard (spike) to a 25 mL portion of sample in a 25-mL mixing cylinder. Stopper and mix thoroughly.
3. Transfer 10 mL to another sample cell (use fill mark) for analysis.

Using AccuVac Ampuls

AccuVac ampuls contain pre-measured powder or liquid in optical-quality glass ampuls.

1. Collect the sample in a beaker or other open container.
2. Place the ampul tip well below the sample surface and break the tip off (see *Figure 6*) against the beaker wall. The break must be far enough below the surface to prevent air from being drawn in as the level of the sample lowers (the AccuVac Breaker may be used instead of breaking the ampul against the beaker side).
3. Invert the ampul several times to dissolve the reagent. Do not place your finger over the broken end; the liquid will stay in the ampul when inverted. Wipe the ampul with a towel to remove fingerprints, etc.
4. Insert the ampul into the instrument and read the results directly.

Figure 6 Using AccuVac Ampuls



Using Reagent Powder Pillows

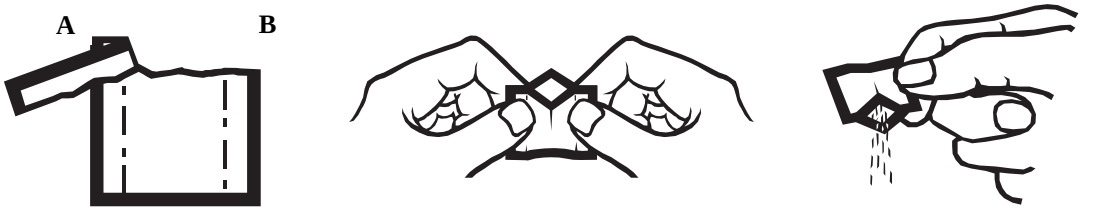
Hach uses dry powdered reagents when possible. This minimizes leakage and deterioration problems. Some powders are packaged in individual, pre-measured, polyethylene "powder pillows" or foil pillows called PermaChem® pillows. Each pillow contains enough reagent for one test. Open the poly powder pillows with nail clippers or scissors; see *Figure 7*.

Figure 7 Opening Powder Pillows

Using PermaChem Pillows

1. Tap the pillow on a hard surface to collect the powdered reagent in the bottom.
2. Tear (or cut) across the top of the pillow, from B to A, holding the pillow away from your face.
3. Using two hands, push both sides toward each other to form a spout.
4. Pour the pillow contents into the sample cell and continue the procedure according to the instructions. Tap the pillow to remove any powder from the corners.

1. Tear **2. Push** **3. Pour**



Reagent and Standard Stability

Hach always strives to make stable formulations and package them to provide maximum protection. Most chemicals and prepared reagents do not deteriorate after manufacture. However, the way they are stored and the packaging can affect how long the reagents are stable. Light, bacterial action, and absorption of moisture and gases from the atmosphere can affect shelf life. Some chemicals may react with the storage container or they may react with other chemicals.

Chemicals supplied with the colorimeter have an indefinite shelf life when stored under average room conditions, unless the packaging says something different. Product labels state any special storage conditions required. Otherwise, store reagents in a cool, dry, dark place for maximum life. It is always good practice to date chemicals when you receive them. Use older supplies first. If in doubt about the reagent shelf life, run a standard to check its effectiveness.

Interferences

Substances in the sample may interfere with a measurement. Hach mentions common interferences in the test procedures. The reagent formulations eliminate many interferences. You can remove others with sample pretreatments described in the procedure.

If you get an unusual answer, a color that you don't expect, or you notice an unusual odor or turbidity, the result may be wrong. Repeat the test on a sample diluted with deionized water; see *Sample Dilution Techniques*. Compare the result (corrected for the dilution) with the result of the original test. If these two are not close, the original result may be wrong and you should make an additional dilution to check the second test (first dilution). Repeat this process until you get the same corrected result twice in a row.

More information about interferences and methods to overcome them is contained in *Standard Additions* of this manual and the *General Introduction* section of APHA Standard Methods. Hach urges the analyst to obtain this book and refer to it when problems are encountered.

One of the greatest aids is knowing what is in the sample. You don't need to know exactly what is in each sample, but be aware of substances that are likely to interfere in the analysis method you use. When using a method, it may be helpful to determine if those interferences are present.

pH Interference

Many of the procedures in this manual only work within a certain pH range. Hach reagents contain buffers to adjust the pH of the typical sample to the correct pH range. However, the reagent buffer may not be strong enough for some samples. This occurs most often with highly buffered samples or samples with extreme sample pH.

The *Sampling and Storage* section of each procedure usually gives the proper pH range for the sample.

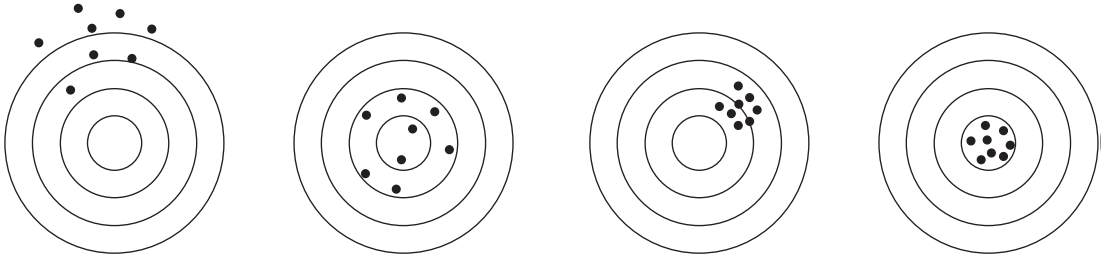
Adjust the sample to the proper pH range before testing. If this information is not given, follow these steps:

1. Measure the pH of your analyzed sample with a pH meter. For measuring Ag^+, K^+ or Cl^- , use pH paper.
2. Prepare a sample using deionized water. Add all reagents called for in the procedure. Timer sequences, etc., may be ignored. Mix well.
3. Measure the pH of the reagent blank with a pH meter.
4. Compare the pH values of your analyzed sample with the reagent blank.
5. If there is little difference in the values of your analyzed sample and the reagent blank, then pH interference is not the problem. Follow the *Accuracy Check* given in the procedure to help identify the problem.
6. If there is a large difference between the value of your analyzed sample and the reagent blank, adjust the sample pH to the value of the reagent blank. Adjust the sample pH to this same pH for all future samples from the same source before analysis. Use the appropriate acid, usually nitric acid, to lower the pH (do not use nitric acid for nitrate or nitrogen testing). Use the appropriate base, usually sodium hydroxide, to raise the pH. Adjust the final result for any dilution caused by adding acid or base; see *Correcting for Volume Additions*.
7. Analyze the sample as before.
8. Some purchased standards may be very acidic and will not work directly with Hach procedures. Adjust the pH of these standards as described above. Adjust the final concentration of the standard for the dilution. The Hach standard solutions suggested in the procedures are formulated so that no pH adjustment is necessary.

Accuracy and Precision

Accuracy is the nearness of a test result to the true value. Precision is how closely repeated measurements agree with each other. Although good precision suggests good accuracy, precise results can be inaccurate (see *Figure 8*). The following paragraphs describe how to improve accuracy and precision of analyses by using Standard Additions.

Figure 8 Precision and Accuracy Illustrated



Standard Additions

Standard Additions is a common technique for checking test results. Other names are “spiking” and “known additions.” The standard additions technique can test for interferences, bad reagents, faulty instruments, and incorrect procedures.

Perform Standard Additions by following the Standard Additions Method section in the procedure under *Accuracy Check*. Follow the detailed instructions given.

If you get about 100% recovery for each addition, everything is working right and your results are correct.

If you don’t get about 100% recovery for each addition, a problem exists. You can tell if you have an interference. Repeat the Standard Additions using deionized water as your sample. If you get about 100% recovery for each addition, you have an interference. If you didn’t get good recoveries with the deionized water, the following checklist may help to find the problem quickly:

1. Check to see that you are following the procedure exactly:

a) Are you using the proper reagents in the proper order? Are you using 10-mL reagents with a 10-mL sample or 25-mL reagents with a 25-mL sample?

b) Are you waiting the necessary time for color to develop?

c) Are you using the correct glassware?

d) Is the glassware clean?

e) Does the test need a specific sample temperature?

f) Is the sample's pH in the correct range?

Hach's written procedure should help you to answer these questions.

2. Check your reagents. Repeat the Standard Additions using new, fresh reagents. If your results are good, the original reagents were bad.

3. If nothing else is wrong, the standard is almost certainly bad. Repeat the Standard Additions with a new standard.

If the check list does not determine the problem, use the decision tree (*Figure 9*) and explanation of each branch, below, to identify the problem.

Branch A

Suppose a single standard addition to the sample did not give the correct concentration increase. A possible cause could be interferences. Other causes include defective reagents, incorrect technique, a defective instrument/apparatus or defective standard used for the standard addition.

If interferences are known or assumed to be absent, proceed to Branch B. If interferences are known to be present, proceed to Branch C.

Branch B

Perform multiple standard additions on a sample of deionized water as in the following example using iron as the analyte of interest:

1. Pour 25 mL of deionized water into a 25-mL sample cell.

2. Add 0.1 mL of a 50-mg/L iron standard solution to a second 25 mL sample of deionized water.

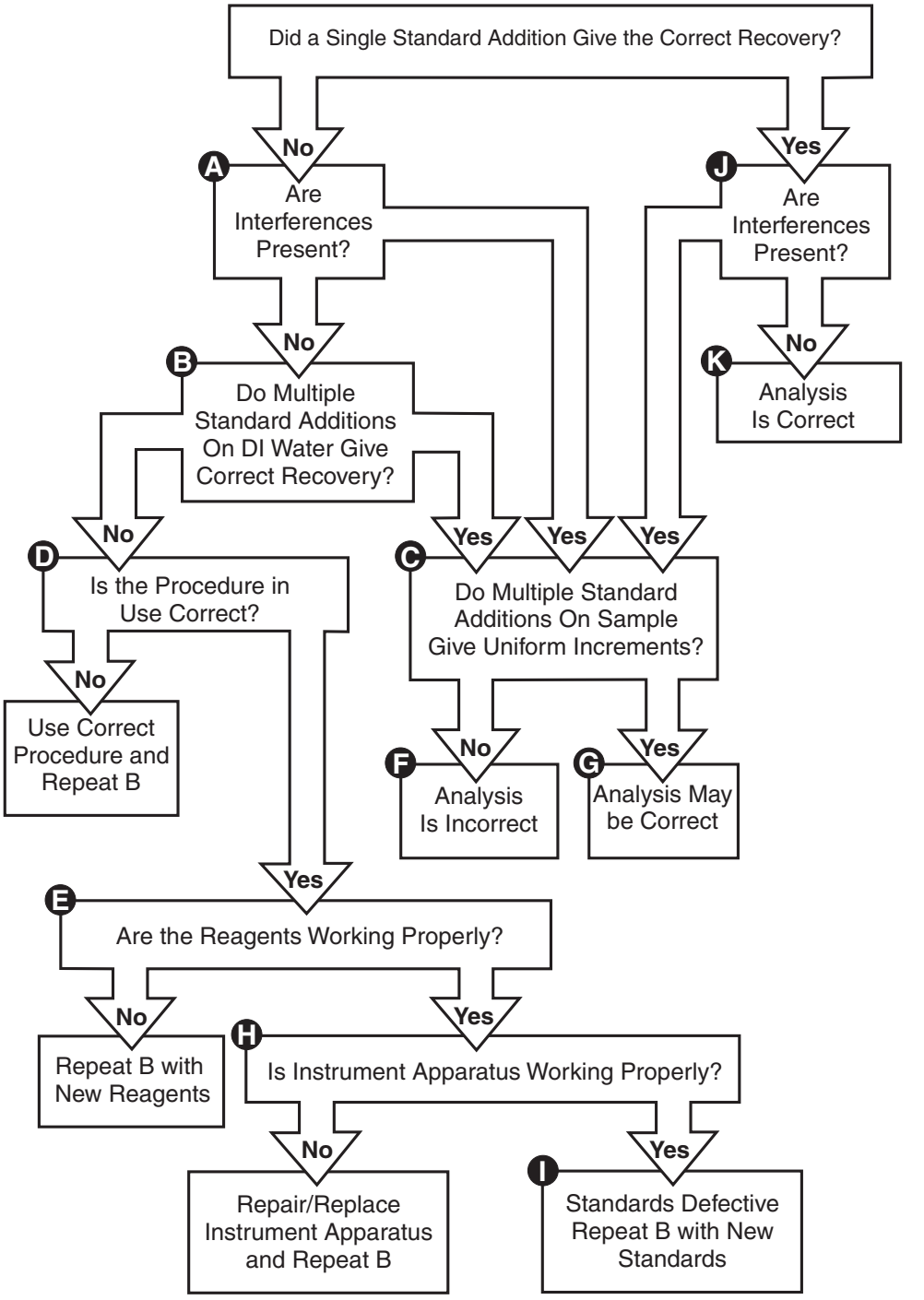
3. Add 0.2 mL of the same standard to a third 25 mL sample of deionized water.

4. Add 0.3 mL of the same standard to a fourth 25 mL sample of deionized water. Analyze all these samples for iron.

5. Tabulate the data as shown below:

mL of Standard Added	mg/L of Standard Added	mg/L of Iron Found
0	0	0
0.1	0.2	0.2
0.2	0.4	0.4
0.3	0.6	0.6

Figure 9 Standard Additions Decision Tree



The data show several points:

- The chemicals, instrument, procedure/technique and standards are working correctly because the iron added to the water sample was completely recovered in the same uniform steps that match the standard addition increments.
- Because iron added to the deionized water was recovered, but iron added to an actual sample was not recovered (Branch A), the sample contains an interference which prevents the test reagents from working properly.
- An iron analysis previously done on the actual sample using this method gave an inaccurate result.

If the results of multiple standard additions give the correct increment for each addition, proceed to Branch C.

If the results of multiple standard additions do not give the correct increment for each addition, go to Branch D.

Branch C

If interfering substances are present, the analysis may be incorrect. However, with multiple standard additions, it may be possible to arrive at an approximate result if the increases are uniform.

Suppose the sample result for iron was 1.0 mg/L. Because interferences may be present, a standard addition of 0.1 mL of a 50 mg/L iron standard to a 25 mL sample is made. The expected increase in the iron concentration is 0.2 mg/L, but the actual increase is 0.1 mg/L. Then 0.2 and 0.3 mL of the same standard are added to two more 25 mL samples and analyzed for iron.

If there is a uniform increase in concentration between each addition (i.e., 0.1 mg/L difference between each addition), use Branch G. If the increase in concentration is not uniform (i.e., 0.1, 0.08, 0.05), go to Branch F.

Branch D

Carefully check the instructions for the test. Make sure to use the correct reagents in the correct order. Be sure the glassware in use is what is required. Be sure time for color development and the sample temperature are as specified. If the procedure technique was incorrect, repeat Branch B. If the procedure was correctly followed, proceed to Branch E.

Branch E

Check the reagent performance. This may be done by obtaining a fresh lot of reagent or by using a known standard solution to run the test. Make sure the color development time given in the procedure is equal to the

time required for the reagent in question. If the reagent(s) is defective, repeat Branch B with new reagents. If the reagents are good, proceed with Branch H.

Branch F

Examples of non-uniform increments between standard additions are shown below.

Example A

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	1.0
0.1	0.2	1.10
0.2	0.4	1.18
0.3	0.6	1.23

Example B

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	0
0.1	0.2	0
0.2	0.4	0.2
0.3	0.6	0.4

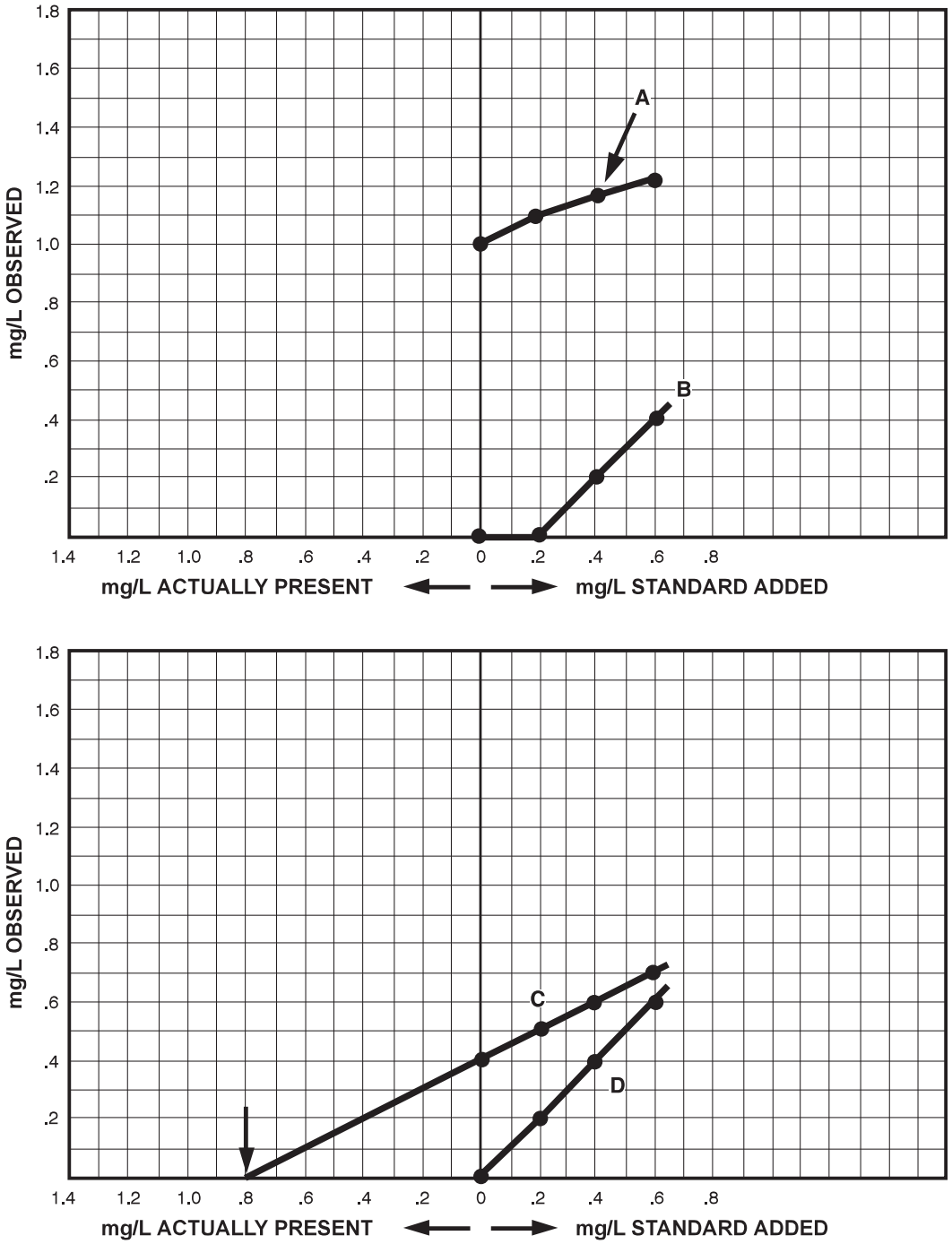
These examples show the effect of interferences on the standard addition. Data plotted on the graph in *Figure 10* for samples A and B show that the four data points do not lie on a straight line.

The plot for sample A illustrates an interference that becomes progressively worse as the concentration of the standard increases. This type of interference is uncommon and may be caused by an error or malfunction of the procedure, reagents or instrument. It is recommended Branch B be performed to verify the supposed interference.

The plot for sample B shows a common chemical interference which becomes less or even zero as the concentration of standard increases. The graph shows the first addition was consumed by the interference and the remaining additions gave the correct increment of 0.2 mg/L.

The apparent interference in Example B could be the result of an error made in the standard addition. Repeat the analysis to see if an error was made during standard addition. If not, the method is not appropriate for the sample matrix. When these two types of interferences occur, try to analyze the sample with a method which uses a different type of chemistry.

Figure 10 Multiple Standard Additions Graph



Branch G

Examples of uniform increments between standard additions are given below.

Example C

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	0.4
0.1	0.2	0.5
0.2	0.4	0.6
0.3	0.6	0.7

The plot for sample C illustrates a common interference with a uniform effect on the standard and the substances in the sample. The four data points form a straight line which may be extended back through the horizontal axis. The point where the line meets the axis can be used to determine the concentration of the substance you are measuring.

In this example, the first analysis gave 0.4 mg/L. After extrapolating the line to the horizontal axis, the graph shows the result should be much closer to the correct result: 0.8 mg/L.

Apparent interferences may also be caused by a defect in the instrument or standards. Before assuming the interference is chemical, check Branch B.

Example D

mL of Standard Added	mg/L Standard Added	mg/L Found
0	0	0
0.1	0.2	0.2
0.2	0.4	0.4
0.3	0.6	0.6

The plot for sample D illustrates a problem for the analyst. The increments are uniform and the recovery of the standard was complete. The result of the first analysis was 0 mg/L and the line extrapolates back through 0 mg/L. If interferences are known to be present, the interferences may be present in an amount equal to the substance in question, preventing the analyst from finding the substance. This would be an uncommon situation.

Branch H

Check operation of the instrument and/or apparatus used to perform the test. Check glassware used in the procedure and make sure it is extremely clean. Dirty pipets and graduated cylinders can cause contamination and will not deliver the correct volume. Check delivery of pipets by using deionized water and a balance; 0.2 mL = 0.2 grams.

If a defect is found in the instrument and/or apparatus, repeat Branch B after repair or replacement. If the instrument and apparatus are working, proceed with Branch I.

Branch I

After determining the procedure, reagents, instrument and/or apparatus are correct and working properly, you may conclude the only possible cause for standard additions not functioning correctly in deionized water is the standard used for performing standard additions. Obtain a new standard and repeat Branch B.

Branch J

If the standard additions gives the correct result, the analyst must then determine if an interfering substance(s) is present. If interfering substances are present, proceed to Branch C. If they are not present, the analysis is correct.

If you still cannot identify the problem, extra help is available. Please call our Technical Support Group at 800-227-4224 (U.S.A.) or 970-669-3050. A representative will be happy to help you.

Method Performance

Estimated Detection Limit

Ranges for chemical measurements have limits. The lower limit is important because it determines whether a measurement is different from zero. Many experts disagree about the definition of this detection limit, and determining it can be difficult. The Code of Federal Regulations (40 CFR, Part 136, Appendix B) provides a procedure to determine the "Method Detection Limit" or MDL. The MDL is the lowest concentration that is different from zero with a 99% level of confidence. A measurement below this MDL may be useful, but there is a greater chance that it is actually zero.

The MDL is not fixed; it varies for each reagent lot, instrument, analyst, sample type, etc. Therefore, a published MDL may be a useful guide, but is only accurate for a specific set of circumstances. Each analyst should determine a more accurate MDL for each specific sample matrix using the same equipment, reagents and standards that will routinely be used for measurements.

Hach provides a value called the Estimated Detection Limit (EDL) for all programs. It is the calculated lowest average concentration in a deionized water matrix that is different from zero with a 99% level of confidence. Specifically, it is the upper 99% confidence limit for zero concentration based on the calibration data used to prepare the pre-programmed calibration curve. **Do not use the EDL as the MDL**. The conditions for MDL determination must be exactly the same as the conditions used for analysis. The EDL may be useful to the analyst as a starting point in determining a MDL or as a way to compare methods. Measurements below the EDL may also be valuable because they can show a trend, indicate the presence of analyte and/or provide statistical data. However, these values have a large uncertainty.

Method Detection Limit (MDL)

This method is in accordance with the USEPA definition in 40 CFR, Part 136, Appendix B (see most current edition).

The USEPA defines the method detection limit (MDL) as the minimum concentration that can be determined with 99% confidence that the true concentration is greater than zero. Since the MDL will vary from analyst to analyst, it is important that analysts determine the MDL based on their unique operating conditions.

The procedure for determining MDL is based on replicate analyses at a concentration 1 to 5 times the estimated detection limit. The MDL value is calculated from the standard deviation of the replicate study results multiplied by the appropriate Student's *t* value for a 99% confidence interval. For this definition, the MDL does not account for variation in sample composition and can only be achieved under ideal conditions.

1. Estimate the detection limit. Use the Hach estimated detection limit (EDL) value stated in the *Method Performance* section of the analysis procedure.

2. Prepare a laboratory standard of the analyte in deionized water which is free of the analyte that is 1 to 5 times the estimated detection limit.

3. Analyze at least seven portions of the laboratory standard and record each result.

4. Calculate the average and standard deviation *(s)* of the results.

5. Compute the MDL using the appropriate Student's *t* value (see table below) and the standard deviation value:

MDL = Student's *t* x *s*

Number of Test Portions	Student's *t* Value
7	3.143
8	2.998
9	2.896
10	2.821

For example:
The EDL for measuring iron using the FerroZine method is 0.003 mg/L. An analyst accurately prepared 1 liter of a 0.010 mg/L (about 3x the EDL) laboratory standard by diluting a 10-mg/L iron standard in iron-free deionized water.

Eight portions of the standard were tested according to the FerroZine method with the following results:

Sample #	Result (mg/L)
1	0.009
2	0.010
3	0.009
4	0.010
5	0.008
6	0.011
7	0.010
8	0.009

Using a calculator program, the average concentration = 0.010 mg/L and the standard deviation (*s*) = 0.0009 mg/L

Based on the USEPA's definition, calculate the MDL as follows:

MDL for FerroZine method = 2.998 (Student's *t*) x 0.0009 (*s*)

MDL = 0.003 mg/L (agrees with initial estimate)

Note: *Occasionally, the calculated MDL may be very different than Hach's estimate of the detection limit. To test how reasonable the calculated MDL is, repeat the procedure using a standard near the calculated MDL. The average result calculated for the second MDL derivation should agree with the initial calculated MDL. Refer to 40 CFR, Part 136, Appendix B (7-1-94), pages 635-637 for detailed procedures to verify the MDL determination.*

Note: *Run a laboratory blank, containing deionized water without analyte, through the test procedure to confirm that the blank measurement is less than the calculated MDL. If the blank measurement is near the calculated MDL, repeat the MDL procedure using a separate blank for analysis for each standard solution portion analyzed. Subtract the average blank measurement from each standard and use the corrected standard values to calculate the average and standard deviation used in the MDL.*

Precision

Every measurement has some degree of uncertainty. Just as a ruler with markings of 0.1 mm leaves some doubt as to the exact length of a measurement, chemical measurements also have some degree of uncertainty. The quality of the entire chemical method determines the precision.

Uncertainty in chemical measurements may be due to systematic errors and/or random errors. A systematic error is a mistake that is always the same for every measurement made. For example, a blank can add to each measurement for a specific compound, giving consistently high results (a positive bias). Random errors are different for every test and add either positive or negative bias. Random errors may be caused by variation in analytical technique and cause response variation. Hach chemists work hard to eliminate systematic errors in Hach procedures using Hach reagents, but response variation occurs in all chemical measurements.

Estimating Precision

The method performance section in each procedure provides an estimate of the procedure's precision. The procedures use a "replicate analysis" estimate, based on real data.

In replicate analysis, a Hach chemist prepares a specific concentration of the analyte in a deionized water matrix. The standard is then analyzed seven individual times with the two reagent lots used in the calibration (14 total samples). A standard deviation of the two sets of seven values is calculated. The larger value is reported in the method. The reported value provides an estimate of the "scatter" of results at a particular point in the calibration curve.

It is important to stress that the estimates are based on a deionized water matrix. Precision on real samples with varying matrices can be quite different than these estimates.

Reagent Blank Correction

The Reagent Blank Correction subtracts the color absorbed when running the test with deionized water instead of sample. The blank value is subtracted from every result to correct for any background color due to reagents.

When using the Reagent Blank Correction feature, the blank correction should be entered before the Standard Adjust feature is used.

To enter a programmed correction for the reagent blank:

1. Run the test using deionized water with each new lot of reagents.
2. Press **READ** to obtain the blank value.
3. Press **SETUP**, scroll to **BLANK** and press **ENTER**. The display will show **BLANK?**.
4. Enter the blank value just read from the instrument.
5. Press **ENTER** to accept the value as the blank to be subtracted from each reading.
6. The display will show 0.00 mg/L (resolution and units vary) and the sample cell icon will be displayed, indicating that the reagent blank feature is enabled and the blank value will be subtracted from each reading. Repeat the reagent blank adjust for each new lot of reagents.

Note: *After entering a reagent blank adjust, the display may flash "limit" when zeroing if the sample used for zeroing has a lower absorbance value than the reagent blank.*

To disable the Reagent Blank adjust feature, press **SETUP**, scroll to **BLANK** and press **ENTER** twice. The concentration readings will be displayed without subtracting the blank. The sample cell icon will no longer appear in the display.

Do not use the Reagent Blank Adjust feature if the procedure uses a reagent blank for zeroing.

Standard Adjust (Adjusting the Standard Curve)

The colorimeter has Hach Programs permanently installed in memory. A program usually includes a pre-programmed calibration curve. Each curve is the result of an extensive calibration performed under ideal conditions and is normally adequate for most testing. Deviations from the curve can occur from using compromised testing reagents, defective sample cells, incorrect test procedure, incorrect technique, or other correctable causes. Interfering substances or other causes may be beyond the analyst's control.

In some situations, using the pre-programmed curve may not be convenient:

a) Running tests where frequent calibration curve checks are required.

b) Testing samples which give a consistent test interference.

Consider the following before adjusting the calibration curve:

1. Will future test results be improved by adjusting the curve?
2. Are interfering substances consistent in all the samples that you will test?

Any precision and test range information provided with the procedure may not apply to an adjusted curve calibration.

You can adjust many of the calibration curves by following the steps found in the test procedures. Working carefully is important. After the adjustment, it is wise to run standard solutions of several concentrations to make sure the adjusted curve is satisfactory. Perform standard additions on typical samples to help determine if the adjusted curve is acceptable.

Think of the standard adjust measurement as a two-step process. First, the instrument measures the sample using the pre-programmed calibration. Second, it multiplies this measurement by an adjustment factor. The factor is the same for all concentrations. The instrument will remember the factor indefinitely and will display the standard adjustment icon when it is used.

Adjust the calibration curve using the reading obtained with a Hach Standard Solution or carefully prepared standard made from a concentrated Hach Standard Solution. It is important to adjust the curve in the correct concentration range. For most purposes, Hach recommends adjusting the curve using a standard concentration that is 70 to 85% of the maximum concentration range of the test.

For example, the Hach pre-programmed method for fluoride has a range of 0-2.0 mg/L F. To adjust the calibration curve, use a standard with a concentration between 1.4-1.6 mg/L. Hach provides a 1.60 mg/L Fluoride Standard Solution (80% of the full range). This is a convenient standard to use for adjusting the calibration curve.

If the range of all your samples is known to be below a concentration that is less than 50% of the full range (50% of 2.0 is 1.0 mg/L), then adjust the standard curve with a standard that is within that range. For example, if all the samples contain 0.6-0.9 mg/L F, you may use a 1.00 mg/L fluoride standard to adjust the curve. You may use the 1.00 mg/L standard because it is closer to the sample range you are working with.

If you are using a Reagent Blank Correction, the blank correction should be entered before the standard curve is adjusted.

To adjust the standard curve:

1. Prepare the standard.
2. Use the standard as the sample in the procedure.
3. When the reading for the standard is obtained, press **SETUP**.
4. Use the arrow keys to scroll to the "**STD**" setup option.
5. Press **ENTER** to activate the standard adjust option.
6. Edit the standard concentration to match that of the standard used.
7. Press **ENTER**. A small plot of a line through a point will be displayed, indicating that the curve has been adjusted with the standard.

Note: *If the attempted correction is outside the allowable adjustment limit, the instrument will beep and flash Ø and the operation will not be allowed.*

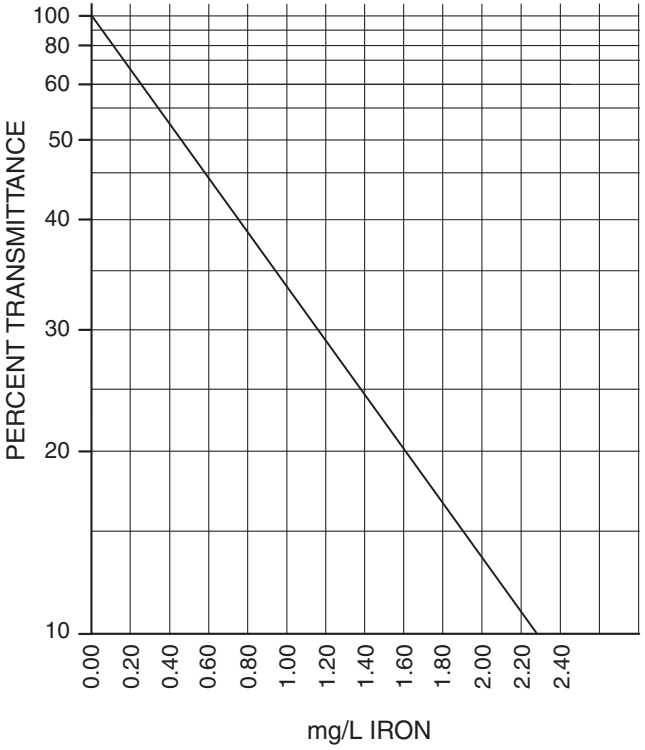
Preparing a User-Entered Calibration Curve

1. Prepare five or more standards of known concentration that cover the expected range of the test. Run tests as described in the procedure on each prepared standard. Pour the customary volume of each known solution into a separate clean sample cell of the type specified for your instrument.
2. Standardize (zero) the instrument using an untreated water sample or a reagent blank, whichever the procedure instructs you to use.
3. Measure and record the absorbance or %T of the known solutions. To use %T vs. concentration see *%T Versus Concentration Calibration*. To use absorbance vs. concentration, see *Absorbance Versus Concentration Calibration*. Or create a user-entered program by storing a custom calibration in the non-volatile memory of the instrument. Refer to the section on entering user-entered programs in the instrument manual.

%T Versus Concentration Calibration

If measuring %T, use semilogarithmic graph paper and plot %T (vertical scale) versus concentration (horizontal scale). In *Figure 11*, iron standard solutions of 0.1, 0.2, 0.4, 0.8, 1.2, 1.6, and 2.0 mg/L were measured on a spectrophotometer at 500 nm using half-inch test tubes. Results were plotted and the calibration table values were extrapolated from the curve (*Table 7*).

Figure 11 Logarithmic Calibration Curve



To convert %T readings to concentration, prepare a table such as *Table 7* and select the appropriate line from the "%T Tens" column and the appropriate column from the %T Units columns. The %T Ten value is the first number of the %T reading and the %T Units value is the second number of the %T reading. For example, if the instrument reading was 46%, the 40 line in the %T Tens column and the 6 column in the %T Units would be selected. The cell where these two intersect (0.78 mg/L) is the iron concentration of the sample.

Table 7 Calibration Table

%T Tens	%T Units									
	0	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
0										
10	2.30	2.21	2.12	2.04	1.97	1.90	1.83	1.77	1.72	1.66
20	1.61	1.56	1.51	1.47	1.43	1.39	1.35	1.31	1.27	1.24
30	1.20	1.17	1.14	1.11	1.08	1.04	1.02	.99	.97	.94
40	.92	.89	.87	.84	.82.	.80	.78	.76	.73	.71
50	.69	.67	.65	.64	.62	.60	.58	.56	.55	.53
60	.51	.49	.48	.46	.45	.43	.42	.40	.39	.37
70	.36	.34	.33	.32	.30	.29	.28	.26	.25	.24
80	.22	.21	.20	.19	.17	.16	.15	.14	.13	.12
90	.11	.09	.08	.07	.06	.05	.04	.03	.02	.01

Absorbance Versus Concentration Calibration

To read concentration values directly from the instrument, create a user-entered program. See the instrument manual for more information.

If absorbance values are measured, plot the results on linear graph paper. Plot the absorbance value on the vertical axis and the concentration on the horizontal axis.

Plot increasing absorbance values from bottom to top. Plot increasing concentration values from left to right. Values of 0.000 absorbance units and 0 concentration will begin at the bottom left corner of the graph. A calibration table can be extrapolated from the curve or the concentration values can be read directly from the graph for determining an equation for the line using the slope and the y-intercept.

USEPA Approved and Accepted Definitions

The United States Environmental Protection Agency (USEPA) establishes limits for maximum contamination levels of certain constituents in water. It also requires that specific methodology be used to analyze for these constituents. These methods originate from several sources. The USEPA has developed some of these methods. In other cases, the USEPA has evaluated and approved methods developed by manufacturers, professional groups and public agencies such as:

- American Public Health Association

- American Water Works Association
- Water Environmental Federation
- American Society for Testing and Materials
- United States Geological Survey
- Associates of Official Analytical Chemists

All USEPA approved methods are cited in the *Federal Register* and compiled in the Code of Federal Regulations. USEPA approved methods may be used for reporting results to the USEPA and other regulatory agencies.

USEPA Accepted

Hach has developed several procedures that are equivalent to USEPA approved methods. Even though minor modifications exist, the USEPA has reviewed and accepted certain procedures for reporting purposes. These methods are not published in the *Federal Register,* but are referenced to the equivalent USEPA method in the procedure.

Digestion

Several procedures require sample digestion. Digestion uses chemicals and heat to break down a substance into components that can be analyzed. This section has three different digestion procedures.

The Hach Digesdahl® system is a process that yields a digest suitable for the determination of metals, total phosphorus and total kjeldahl nitrogen (TKN). It is rapid, convenient and the method of choice for digesting most samples analyzed by Hach methods.

For USEPA reporting purposes, USEPA-approved digestions are required. USEPA presents two digestions (mild and vigorous) for metals analysis. These are much more inconvenient and time consuming compared to the Hach Digesdahl system. Other digestion procedures are required for phosphorus and TKN.

EPA Mild Digestion with Hot Plate for Metals Analysis Only

1. Acidify the entire sample at the time of collection with concentrated nitric acid by adding 5 mL of acid per liter (or quart) of sample.

2. Transfer 100 mL of well-mixed sample to a beaker or flask. Add 5 mL of distilled 1:1 hydrochloric acid (HCl).

3. Heat using a steam bath or hot plate until the volume has been reduced to 15-20 mL. Make certain the sample does not boil.

4. After this treatment, the sample may be filtered to remove any insoluble material.

5. Adjust the digested sample to pH 4 by drop-wise addition of 5.0 N Sodium Hydroxide Standard Solution. Mix thoroughly and check the pH after each addition.

6. Quantitatively transfer the sample with deionized water to a 100-mL volumetric flask and dilute to volume with deionized water. Continue with the procedure. This mild digestion may not suffice for all sample types. A reagent blank also should be carried through the digestion and measurement procedures.

EPA Vigorous Digestion with Hot Plate for Metals Analysis Only

A vigorous digestion can be followed to ensure all organo-metallic bonds are broken.

1. Acidify the entire sample with redistilled 1:1 Nitric Acid Solution to a pH of less than two. Do not filter the sample before digestion.

2. Transfer an appropriate sample volume (see *Table 8*) into a beaker and add 3 mL of concentrated redistilled nitric acid.

3. Place the beaker on a hot plate and evaporate to near dryness, making certain the sample does not boil.

4. Cool the beaker and add another 3 mL of the concentrated redistilled nitric acid.

5. Cover the beaker with a watch glass and return it to the hot plate. Increase the temperature of the hot plate so that a gentle reflux occurs. Add additional acid, if necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change color or appearance with continued refluxing).

6. Again, evaporate to near dryness (do not bake) and cool the beaker. If any residue or precipitate results from the evaporation, add redistilled 1:1 hydrochloric acid (5 mL per 100 mL of final volume). See *Table 8*.

7. Warm the beaker. Add 5 mL of 5.0 N sodium hydroxide and quantitatively transfer the sample with deionized water to a volumetric flask. See *Table 8* below for the suggested final volume.

8. Adjust the sample to pH 4 by drop-wise addition of 5.0 N Sodium Hydroxide Standard Solution; mix thoroughly and check the pH after each addition. Dilute to volume with deionized water. Multiply the result by the correction factor in *Table 8*. A reagent blank also should be carried through the digestion and measurement procedures.

Table 8 Vigorous Digestion Volumes

Expected Metal Concentration	Suggested Sample Vol. for Digestion	Suggested Volume of 1:1 HCl	Suggested Final Volume After Digestion	Correction Factor
1 mg/L	50 mL	10 mL	200 mL	4
10 mg/L	5 mL	10 mL	200 mL	40
100 mg/L	1 mL	25 mL	500 mL	500

General Digesdahl Digestion (Not USEPA accepted)

Many samples may be digested using the Digesdahl Digestion Apparatus (Cat. No. 23130). It is designed to digest many types of samples such as oils, wastewater, sludges, feeds, grains, plating baths, food, and soils. In this procedure the sample is oxidized by a mixture of sulfuric acid and hydrogen peroxide. Digestion of a dry sample requires less than ten minutes, while liquid samples require about 1 minute/mL. The digestion is done in a special flat-bottomed 100-mL volumetric flask. Aliquots (sample portions) are taken for analysis using colorimetric methods.

Procedures for digestion and using the Digesdahl Digestion Apparatus are based on the type and form of the sample, and are found in the Digesdahl Digestion Apparatus Instruction Manual, which is included with each Digesdahl Digestion Apparatus.

Distillation

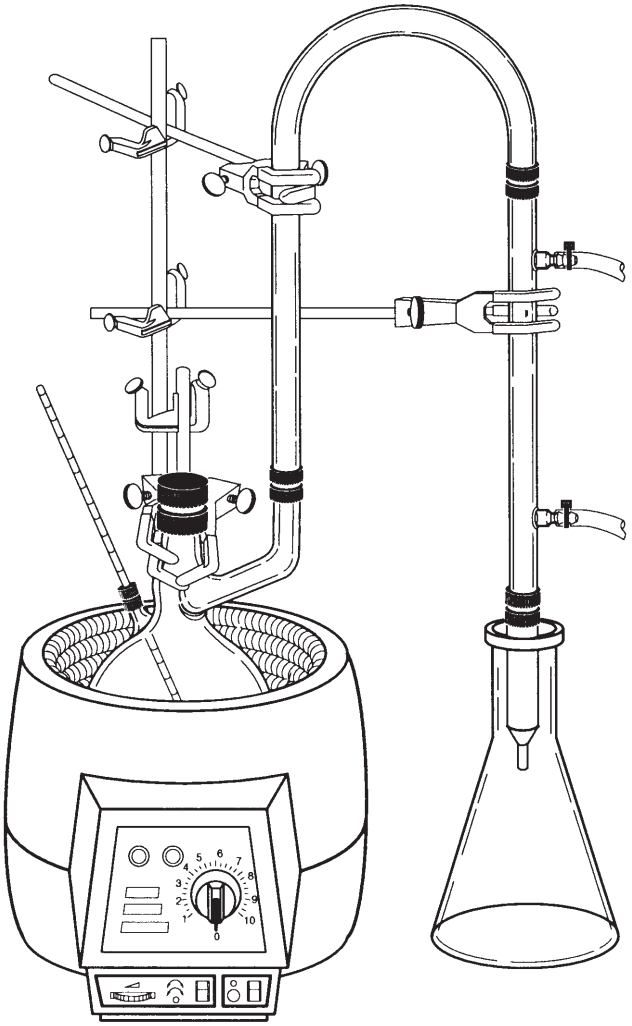
Distillation is an effective way of separating chemical components for analysis. The Hach Distillation Apparatus (see *Figure 12*) is adapted easily for many test needs and is suitable for water and wastewater samples. Sample distillations are easy and safe to perform.

Applications for the General Purpose Distillation Apparatus include:

- fluoride
- albuminoid nitrogen
- ammonia nitrogen
- phenols
- selenium
- volatile acids

Arsenic and cyanide require special glassware sets in addition to the General Purpose Set (the Arsenic Distillation Apparatus and the Cyanide Distillation Apparatus). All connecting glassware is manufactured with threaded connectors for ease and safety. The General Purpose Heater provides efficient heating and the Support Apparatus anchors the glassware.

Figure 12 General Purpose Distillation Apparatus with Heater and Support Apparatus



SECTION 3 WASTE MANAGEMENT AND SAFETY

Waste Management

This section provides guidelines for laboratory waste management. It should assist you in complying with USEPA regulations governing waste management. It summarizes basic requirements, but does not contain all USEPA regulations. It does not relieve people from complying with all regulations contained in the Code of Federal Regulations. Regulations change regularly and additional state and local laws may apply to your waste. Each waste generator is responsible for knowing and obeying the laws that apply to them.

Waste Minimization

Waste minimization is the foundation of good waste management. Minimizing waste greatly reduces the disposal problems and expense. If possible, try to generate less waste rather than recycle or re-use it. For laboratories, ways to reduce waste include:

- Use the smallest sample size possible.
- Choose methods that use non-hazardous or “less” hazardous reagents when possible.
- Buy chemicals in small quantities which will be used before they expire. This eliminates disposal of outdated materials.
- Clean glassware and laboratory apparatus with non-hazardous soaps when possible, rather than solvents or acids which may be hazardous.

Regulatory Overview

Federal waste disposal regulations were issued in accordance with the Resource Conservation and Recovery Act (RCRA). They are given in Title 40 Code of Federal Regulations (CFR) part 260. The Act controls all forms of solid waste disposal and encourages recycling and alternative energy sources. The major emphasis is controlling hazardous waste disposal. The regulations create a system to identify wastes and track waste generation, transport, and ultimate disposal. Each facility involved in managing hazardous waste must be registered with the USEPA. This includes the generator, transporters, and treatment, storage, and disposal facilities (TSDF).

Under federal regulations, there are three categories of generators with increasingly more strict regulation for larger quantity generators. The categories are based on the amount of hazardous waste generated in any given month.

The categories are as follows:

- Conditionally Exempt Small Quantity Generator - less than 100 kg (220 lb.) per month
- Small Quantity Generator - between 100 kg (220 lb.) and 1,000 kg (2,200 lb.) per month
- Large Quantity Generator - greater than 1,000 kg (2,200 lb.) per month

Note: *If a laboratory generates acutely hazardous waste (as defined on 40 CFR 261) or accumulates more than a certain amount of waste, the facility may be moved into a larger generator status. Check with your environmental compliance manager or state and local officials to determine which category your facility is in.*

Hazardous Waste Definition

For regulatory purposes, a "hazardous waste" is a material which is subject to special laws by the USEPA under 40 CFR 261. In addition, many states or local authorities regulate additional materials as hazardous waste. Be aware that many very toxic compounds are not regulated by this definition of hazardous waste. However, improper management or disposal of these compounds may lead to legal problems under other laws such as CERCLA (Superfund) or common law torts.

The 40 CFR 261 defines a hazardous waste as a solid waste which is not excluded from regulation and meets any of the following criteria:

- It is a discarded commercial chemical product, off-specification species, container residue, or spill residue of materials specifically listed in 40 CFR 261.33;
- It is a waste from a specific source listed in 40 CFR 261.32;
- It is a waste from a non-specific source listed in 40 CFR 261.31; or
- It displays any of the following characteristics of hazardous waste defined in 40 CFR 261.20-24:
    - ignitability
    - corrosivity
    - reactivity
    - toxicity

There are many exceptions to these regulations, and each generator should review the regulations and determine if they are excluded from the regulations.

Characteristic Hazardous Waste Codes

Hazardous wastes are categorized by specific codes assigned in 40 CFR 261.20-261.33. These codes will help you identify hazardous waste. The generator is responsible for making the actual waste code determination.

Selected characteristic waste codes for chemicals which may be generated using Hach methods for water analysis are given in the following table. A complete list of waste codes is found in 40 CFR 261.24.

USEPA Code	Characteristic	CAS No.	Regulatory Level (mg/L)
D001	Ignitability	na	na
D002	Corrosivity	na	na
D003	Reactivity	na	na
D004	Arsenic	6440-38-2	5.0
D005	Barium	6440-39-3	100.0
D018	Benzene	71-43-2	0.5
D006	Cadmium	7440-43-9	1.0
D022	Chloroform	67-66-3	6.0
D007	Chromium	7440-47-3	5.0
D008	Lead	7439-92-1	5.0
D009	Mercury	7439-97-6	0.2
D010	Selenium	7782-49-2	1.0
D011	Silver	7440-22-4	5.0

How to Determine if Waste is Hazardous

Federal laws do not require you to test a material to decide if it is a hazardous waste. You may apply product knowledge to decide if a material is hazardous. Often, information on a material safety data sheet (MSDS) is enough to decide. If the product is specifically listed in the regulation, it is a hazardous waste.

You also need to decide if it has any characteristics of a hazardous waste. Physical information on the MSDS may help you decide. If the flash point is below 60 °F (15 °C) or is classified by DOT as an oxidizer, the material may be ignitable. If the pH of the material is ≤2 or ≥12.5, the material may be corrosive. If the material is unstable, reacts violently with water, or may generate toxic gases, vapors, or fumes when mixed with water, it may be reactive.

Use the chemical composition data to decide if a material is toxic. This decision is based on the concentration of certain contaminants (heavy metals and a number of organic compounds). If the waste is a liquid, compare the concentration of the contaminants in the liquid to the concentrations listed in 40 CFR 261.24. If the waste is a solid, analyze the sample by the Toxicity Characteristic Leachability Procedure (TCLP) and compare the results to the concentration listed in the 40 CFR 261.24. Levels above the threshold amount listed in the table are hazardous.

See "Sections of the MSDS" on page 63. describing the MSDS for help in finding information for making hazardous waste determinations.

Examples of Hazardous Waste

A number of chemicals used in and final solutions created from Hach procedures are hazardous wastes when they are disposed. In addition, substances in the sample matrix may be a hazardous waste. Sometimes, reagents which would be hazardous are neutralized or changed during the analytical procedure. In that case, the final solutions are not regulated. Finally, many reagents and final solutions may be non-regulated. The generator must either use their knowledge of the materials used or conduct analytical tests to determine if the final material is a hazardous waste.

Examples of tests using Hach reagents that generate hazardous waste include those containing mercury or mercury compounds such as COD tests or Nessler's reagent. Conversely, a test using Hach reagents such as ManVer 2 Hardness Indicator Powder Pillows and EDTA Titration Cartridges do not produce a hazardous waste unless the sample contains a hazardous substance.

Hazardous Waste Disposal

Hazardous waste must be managed and disposed of according to federal, state, and local regulations. The waste generator is responsible for making hazardous waste determinations. Analysts should check with the facility's environmental compliance people for specific instructions.

Hazardous wastes should be handled by treatment, storage, and disposal facilities (TSDF) that have USEPA permits. In some cases, the generator may treat the hazardous waste. In most cases, a permit from the USEPA is required to treat hazardous waste. Laboratories are not exempt from these regulations. If your facility is a "Conditionally Exempt Small Quantity Generator," special rules may apply. Check 40 CFR 261 to determine if have to comply with all the laws.

The most common allowed treatment is elementary neutralization. This refers to neutralizing wastes that are hazardous only because they are corrosive or are listed only for that reason. Neutralize acidic solutions by adding a base such as sodium hydroxide; neutralize basic solutions by

adding an acid such as hydrochloric acid. Slowly add the neutralizing agent while stirring. Monitor the pH. When it is at or near 7, the material is neutralized and may be flushed down the drain. Many wastes generated from Hach procedures may be treated in this manner.

Other chemical or physical treatments such as cyanide destruction or evaporation may require a permit. Check with your environmental department or local regulators to determine which rules apply to your work facility.

Laboratory chemicals may be mixed and disposed of with other hazardous wastes generated at your facility. They may also be accumulated in accordance with 40 CFR 262.34 satellite accumulation rules. After collection they may be disposed of in a "labpack." A number of environmental and hazardous waste companies offer labpacking services. They will inventory, sort, pack, and arrange proper disposal for hazardous waste. Find companies offering these services in the Yellow Pages under "Waste Disposal - Hazardous" or contact state and local regulators for assistance.

Management of Specific Wastes

Hach has several documents to assist customers in managing waste generated from our products. You can obtain the following documents by calling 1-800-227-4224 or 970-669-3050 and requesting the literature codes given:

Literature Code	Title
1321	Waste Reduction: A Primer
9323	Mercury Waste Disposal Firms
9325	COD Waste Management
9326	COD Heavy Metal Total Concentrations

Special Considerations for Cyanide-Containing Materials

Several procedures in this manual use reagents that contain cyanide compounds. These materials are regulated as reactive (D003) waste by the Federal RCRA. Waste disposal instructions provided with each procedure tell you how to collect these materials for proper disposal. It is imperative that these materials be handled safely to prevent the release of hydrogen cyanide gas (an extremely toxic material with the smell of bitter almonds). Most cyanide compounds are stable and can be safely stored for disposal in highly alkaline solutions (pH >11) such as 2 N sodium hydroxide. Never mix these wastes with other laboratory wastes that may contain lower pH materials such as acids or even water.

If a cyanide-containing compound is spilled, you must be careful not to be exposed to hydrogen cyanide gas. Take the following steps to destroy the cyanide compounds in an emergency:

a) Use a fume hood, supplied air or self-contained breathing apparatus.

b) While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and either calcium hypochlorite or sodium hypochlorite (household bleach).

c) Add an excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.

d) Neutralize the solution and flush it down the drain with a large amount of water. If the solution contains other regulated materials such as chloroform or heavy metals, it may still need to be collected for hazardous waste disposal. Never flush hazardous wastes down the drain.

Resources

Many sources of information on proper waste management are available. The USEPA has a hotline number for questions about the Resource Conservation and Recovery Act (RCRA). The RCRA Hotline number is 1-800-424-9346. You may also get a copy of the appropriate regulations. Federal hazardous waste regulations are found in 40 CFR 260- 99. Obtain this book from the U.S. Government Printing Office or a number of other vendors. Other documents which may be helpful to the laboratory hazardous waste manager include:

1. Task Force on Laboratory Waste Management. *Laboratory Waste Management, A Guidebook*; American Chemical Society, Department of Government Relations and Science Policy: Washington, DC 1994.

2. Task Force on Laboratory Waste Management. *Waste Management Manual for Laboratory Personnel*; American Chemical Society, Department of Government Relations and Science Policy: Washington, DC 1990.

3. Task Force on Laboratory Waste Management. *Less is Better*; 2nd ed.; American Chemical Society, Department of Government Relations and Science Policy: Washington, DC 1993.

4. Committee on Chemical Safety. *Safety in Academic Chemistry Laboratories*, 5th ed.; American Chemical Society: Washington, DC, 1990.

5. Armour, Margaret-Ann. *Hazardous Laboratory Chemicals Disposal Guide*; CRC Press: Boca Raton, FL, 1991.

6. *Environmental Health and Safety Manager's Handbook*; Government Institutes, Inc.: Rockville, MD, 1988.

7. Lunn, G.; Sansone, E.B. *Destruction of Hazardous Chemicals in the Laboratory*; John Wiley and Sons: New York, 1990.

8. National Research Council. *Prudent Practices for Disposal of Chemicals from Laboratories*; National Academy Press: Washington, DC, 1983.

9. National Research Council. *Prudent Practices for Handling Hazardous Chemicals in Laboratories*; National Academy Press: Washington, DC, 1981.

10. Environmental Protection Agency, Office of Solid Waste and Emergency Response. *The RCRA Orientation Manual*; U.S. Government Printing Office: Washington, DC, 1991.

11. Environmental Protection Agency, Office of Solid Waste and Emergency Response. *Understanding the Small Quantity Generator Hazardous Waste Rules: A Handbook for Small Business*; U.S. Government Printing Office: Washington, DC, 1986.

Material Safety Data Sheets

Material safety data sheets (MSDS) describe the hazards of chemical products. This section describes the information provided on a Hach MSDS and how to locate important information for safety and waste disposal. The information provided on the MSDS applies to the product as sold by Hach. The properties of any mixtures obtained by using this product will be different.

How to Obtain an MSDS

Hach ships an MSDS to each customer with the first order of any chemical product. A new MSDS may be sent when the information on the data sheet is updated. Please review all new MSDS's for new information. If you need another copy of an MSDS, simply call 1-800-227-4227.

Sections of the MSDS

Each MSDS has ten sections. The sections and the information found in them are described below.

Header Information

The Hach catalog number, MSDS date, change number, company address and telephone number, and emergency telephone numbers are listed at the top of the MSDS.

1 Product Identification

This section contains:

- Hach product name
- Chemical Abstract Services (CAS) number
- Chemical name
- Chemical formula, if appropriate
- Chemical family to which the material belongs

2 Ingredients

This section lists each component in the product. It contains the following information for each component:

- PCT: Percent by weight of this component
- CAS NO.: Chemical Abstract Services (CAS) registry number for this component
- SARA: Superfund Amendments and Reauthorization Act, better known as the "Community Right to Know Law" tells you if the component is listed in SARA 313. If the component is listed and you use more than the amount listed, you must report this to the USEPA every year.
- TLV: Threshold Limit Value. The maximum airborne concentration for an 8 hour exposure that is recommended by the American Conference of Governmental Industrial Hygienists (ACGIH).
- PEL: Permissible Exposure Limit. The maximum airborne concentration for an 8 hour exposure that is regulated by the Occupational Safety and Health Administration (OSHA).
- HAZARD: Physical and health hazards of the component are explained.

3 Physical Data

The physical properties of the product are given in this section. They include the physical state, color, odor, solubility, boiling point, melting point, specific gravity, pH, vapor density, evaporation rate, corrosivity, stability, and storage precautions.

4 Fire, Explosion Hazard And Reactivity Data

This section contains the flash point and flammable limits of the material. It also includes how to fight fires if the material catches on fire. Key terms in this section include:

- Flashpoint: The temperature at which a liquid will give off enough flammable vapor to ignite.
- Flammability and ignitability are usually defined by the flash point.
- Lower Flammable Limit (LFL or LEL): The lowest concentration that will produce a fire or flash when an ignition source is present.
- Upper Flammable Limit (UFL or UEL): The vapor concentration in air above which the concentration is too rich to burn.
- NFPA Codes: The National Fire Protection Association (NFPA) has a system to rate the degree of hazards presented by a chemical. These codes are usually placed in a colored diamond. The codes range from 0 for minimal hazard to 4 for extreme hazard. They are grouped into the following hazards: health (blue), flammability (red), reactivity (yellow), and special hazards (white).

5 Health Hazard Data

This section describes different ways the chemical can enter your body (ingestion, inhalation, skin contact). It also gives acute (immediate) and chronic (long-term) health effects. If the material causes cancer or genetic damage, it is identified in this section.

6 Precautionary Measures

This section contains special precautions for the material. These may include special storage instructions, handling instructions, conditions to avoid, and protective equipment required to use this material safely.

7 First Aid

First aid instructions for exposures to the chemical are given in this section. Be sure to read this section before inducing vomiting in a victim. Some chemicals are better treated by not inducing vomiting. Seek prompt medical attention for all chemical exposures.

8 Spill And Disposal Procedures

This section tells about safe work practices for cleaning up and disposing of spilled material. Please refer to the Waste Management section of this manual. Final determination of proper and legal disposal options is the responsibility of the waste generator. Be sure you know the federal, state, and local laws that apply to your facility.

9 Transportation Data

Domestic and International shipping information is provided in this section. It gives shipping name, hazard class, and ID number of the product.

10 References

This section lists the reference materials used to write the MSDS.

Following the Reference section, the product is listed as having SARA 313 chemicals or California Proposition 65 List Chemicals, if applicable. Also found here is any special information about the product.

Safety

Safety is the responsibility of each person performing analytical procedures. Because many of the procedures in this methods manual use potentially hazardous chemicals and equipment, it is important to prevent accidents by practicing good laboratory techniques. The following guidelines apply to water analysis. These guidelines do not cover every aspect of safety, but they are important for preventing injuries.

Material Safety Data Sheet

A material safety data sheet (MSDS) comes with the first shipment of all products. The MSDS provides environmental and safety information about the products. Always read the MSDS before using a new product.

Reading Labels Carefully

Read each reagent label carefully. Pay particular attention to the precautions given. Never remove or block the label on a reagent container while it contains reagent. Do not put a different reagent into a labeled container without changing the label. When preparing a reagent or standard solution, label the container clearly. If a label is hard to read, re-label promptly according to your facility's hazard communication program.

Warning labels also appear on some of the apparatus used with the test procedures. The protective shields with the COD Reactor and the Digesdahl Digestion Apparatus point out potential hazards. Be sure these shields are in place during use and observe the precautions on the label.

Protective Equipment

Use the right protective equipment for the chemicals and procedures. The MSDS contains this information. Protective equipment may include:

- Eye protection such as safety glasses or goggles to protect from flying objects or chemical splashes.
- Gloves to protect skin from toxic or corrosive materials, sharp objects, very hot or very cold materials, or broken glass. Use tongs or finger cots when transferring hot apparatus.

- Laboratory coats or splash aprons to protect skin and clothing from splashes.
- Footwear to protect feet from spills. Open toed shoes should not be worn in chemistry settings.
- Respirators may be needed to protect you from breathing toxic vapors if adequate ventilation, such as fume hoods, are not available.
- Use fume hoods as directed by the procedure or as recommended in the MSDS.
- For many procedures, adequate ventilation is enough. Be sure there is enough fresh air and air exhaust to protect against unnecessary exposure to chemicals.

First Aid Equipment and Supplies

Most first aid instructions for chemical splashes in eyes or on skin call for thorough flushing with water. Laboratories should have eyewash and shower stations. For field work, carry a portable eyewash unit. Laboratories should also have appropriate fire extinguishers and fume hoods.

General Safety Rules

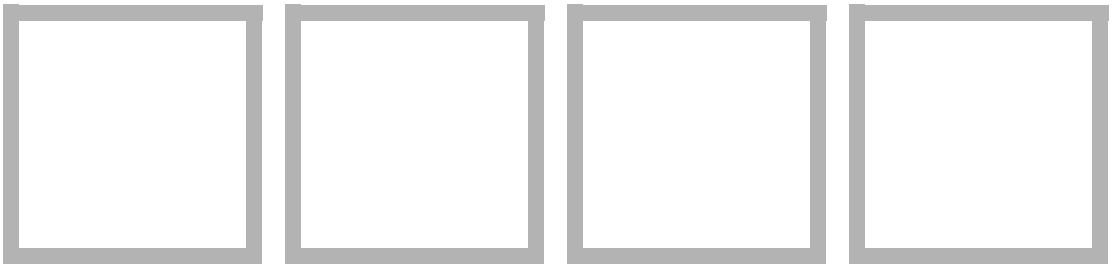
Follow these rules to make work with toxic and hazardous chemicals safer:

1. **Never** pipet by mouth. Always use a mechanical pipet or pipet bulb to avoid ingesting chemicals.
2. Follow test procedures carefully and observe all precautionary measures. Read the entire procedure carefully before beginning.
3. Wipe up all spills promptly. Get proper training and have the right response equipment to clean up spills. See your safety director for more information.
4. **Do not** smoke, eat, or drink in an area where toxic or irritating chemicals are used.
5. Use reagents and equipment only as directed in the test procedure.
6. **Do not** use damaged labware and broken equipment.
7. Minimize all chemical exposures. **Do not** breathe vapors or let chemicals touch your skin. Wash your hands after using chemicals.
8. Keep work areas **neat** and **clean**.

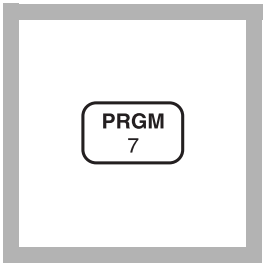
9. **Do not** block exits or emergency equipment.

OSHA Chemical Hygiene Plan

The Occupational Safety and Health Administration (OSHA) enforces laws about the control exposure to hazardous chemicals in laboratories. These regulations are in Title 29 CFR 1910.1450. They apply to all employers who use hazardous chemicals. They require employers to develop and use a written Chemical Hygiene Plan and appoint a qualified person as the Chemical Hygiene Officer.



Aluminon Method*



1. Enter the stored program number for aluminum (Al).

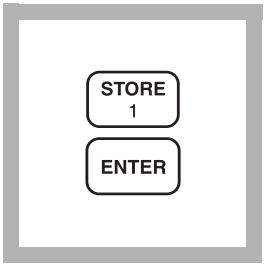
Press: **PRGM**

The display will show:

PRGM ?

Note: *Adjust the pH of stored samples before analysis.*

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

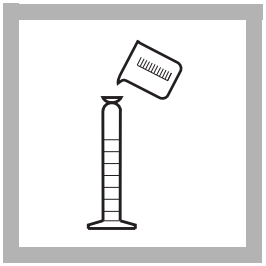


2. Press: **1 ENTER**

The display will show **mg/L, Al** and the **ZERO** icon**.**

Note: *Total aluminum determination requires a digestion prior to analysis (see Section 2).*

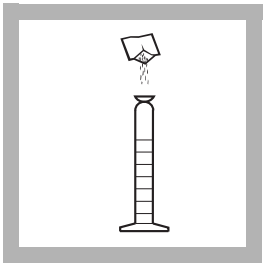
Note: *For alternate form (Al_2O_3), press* **CONC**.



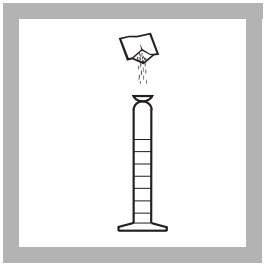
3. Fill a 50-mL graduated mixing cylinder to the 50-mL mark with sample.

Note: *Rinse cylinder with 1:1 Hydrochloric Acid and deionized water before use to avoid errors due to contaminants absorbed on the glass.*

Note: *Sample temperature must be 20-25 °C (68-77 °F) for accurate results.*



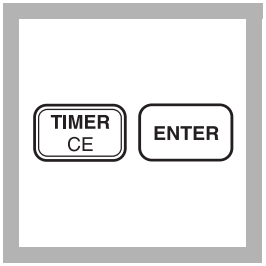
4. Add the contents of one Ascorbic Acid Powder Pillow. Stopper. Invert several times to dissolve powder.



5. Add the contents of one AluVer® 3 Aluminum Reagent Powder Pillow. Stopper.

Note: *A red-orange color develops if aluminum is present.*

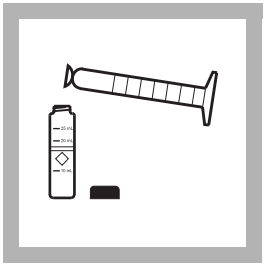
Note: *Inconsistent results will occur if any powder is undissolved.*



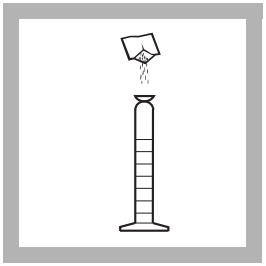
6. Press:

TIMER ENTER

A three-minute reaction period will begin. Invert the cylinder repeatedly for the three minutes.

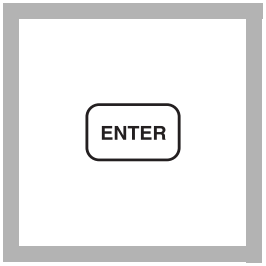


7. Pour 25 mL of mixture into a 25-mL sample cell (the prepared sample).



8. Add the contents of one Bleaching 3 Reagent Powder Pillow to the remaining 25 mL in the mixing graduated cylinder (the blank). Stopper the cylinder.

* Adapted from *Standard Methods for the Examination of Water and Wastewater*

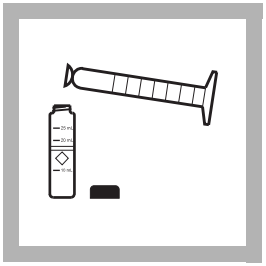


9. The display will show: **00:30 Timer 2**

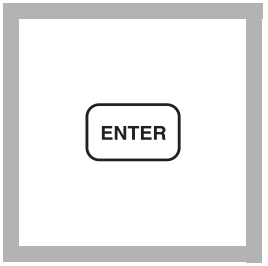
Press: **ENTER**

A thirty-second reaction period will begin. Vigorously shake the cylinder for the 30-second period.

Note: *This solution should turn a light to medium orange upon bleaching. It will not become colorless.*



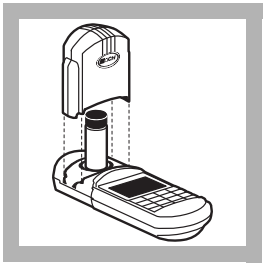
10. Pour the 25 mL of mixture in the cylinder into a second 25-mL sample cell (the blank).



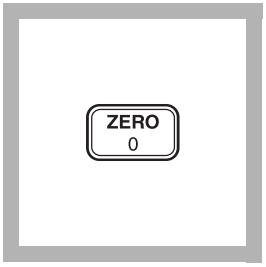
11. The display will show: **15:00 TIMER 3**

Press: **ENTER**

A 15-minute reaction period will begin.



12. Within three minutes after the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

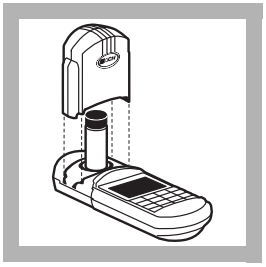


13. Press: **ZERO**

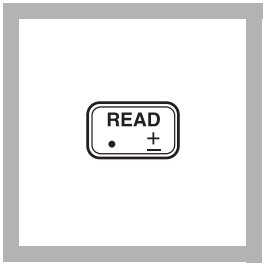
The cursor will move to the right, then the display will show:

0.000 mg/L Al

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



14. Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



15. Press: **READ**

The cursor will move to the right, then the result in mg/L aluminum will be displayed.

Note: *Clean the graduated cylinder and sample cells with soap and brush immediately following the test.*

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in a clean glass or plastic container. Preserve the sample by adjusting the pH to 2 or less with nitric acid (about 1.5 mL per liter). Preserved samples can be stored up to six months at room temperature. Before analysis, adjust the pH to 3.5–4.5 with 5.0 N Sodium Hydroxide. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1* for more information.

Accuracy Check

Standard Additions Method

a) Snap the neck off an Aluminum Voluette Ampule Standard Solution, 50 mg/L as Al.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to three 50-mL samples. Swirl gently to mix. Also prepare a sample without any standard added (the unspiked sample).

c) Analyze each sample as described above. The aluminum concentration should increase 0.1 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

Standard Solution Method

Prepare a 0.40-mg/L aluminum standard solution by pipetting 1.00 mL of Aluminum Standard Solution, 100 mg/L as Al^{3+}, into a 250-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution immediately before use. Perform the aluminum procedure as described above. The mg/L Al reading should be 0.40 mg/L Al.

Or, using the TenSette Pipet, add 0.8 mL of solution from an Aluminum Voluette Ampule Standard Solution (50 mg/L as Al) into a 100-mL volumetric flask. Dilute to volume with deionized water. Prepare this standard immediately before testing and use as the sample.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.40 mg/L Al and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.013 mg/L Al.

Estimated Detection Limit

The estimated detection limit for program #1 is 0.013 mg/L Al. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Acidity interferes at greater than 300 mg/L as $CaCO_3$. Treat samples with greater than 300 mg/L acidity as $CaCO_3$ as follows: **1.** Add one drop of m-Nitrophenol Indicator Solution to the sample taken in Step 3. **2.** Add one drop of 5.0 N Sodium Hydroxide Standard Solution. Stopper the cylinder. Invert to mix. Repeat as often as necessary until the color changes from colorless to yellow. **3.** Add one drop of 5.25 N Sulfuric Acid Standard Solution to change the solution from yellow back to colorless. Continue with the test.
Alkalinity	1000 mg/L as $CaCO_3$. Eliminate interferences from higher alkalinity concentrations using the following pretreatment: **1.** Add one drop of m-Nitrophenol Indicator Solution to the sample taken in Step 3. A yellow color indicates excessive alkalinity. **2.** Add one drop of 5.25 N Sulfuric Acid Standard Solution. Stopper the cylinder. Invert to mix. If the yellow color persists, repeat until the sample becomes colorless. Continue with the test.
Calcium	Does not interfere.
Fluoride	Interferes at all levels. See graph below.
Iron	Greater than 20 mg/L.
Phosphate	Greater than 50 mg/L.
Polyphosphate	Polyphosphate interferes at all levels by causing negative errors and must not be present. Before running the test, polyphosphate must be converted to orthophosphate by acid hydrolysis as described under the phosphorus procedures.

Fluoride interferes at all levels by complexing with aluminum. The actual aluminum concentration can be determined using the Fluoride Interference Graph when the fluoride concentration is known. To use the fluoride interference graph:

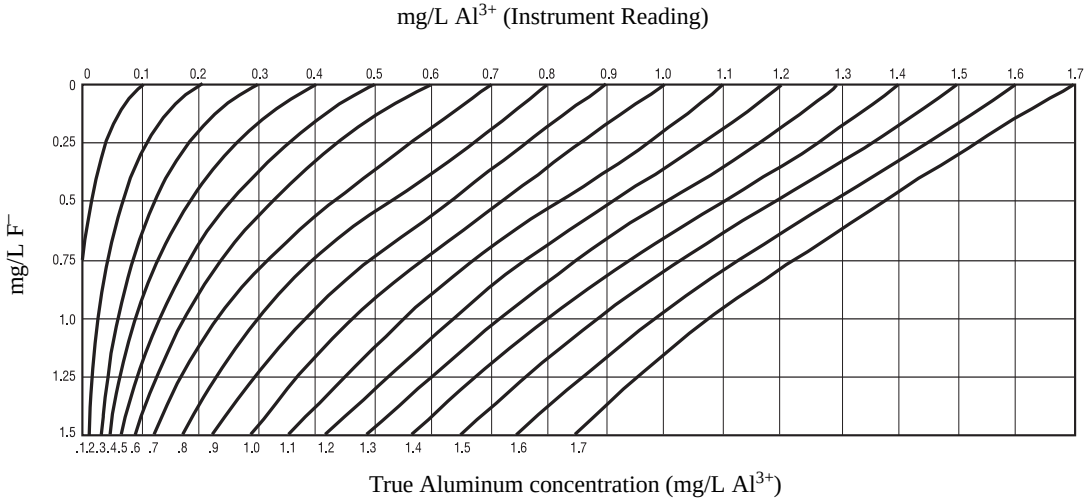
1. Select the vertical grid line along the top of the graph that represents the aluminum reading obtained in Step 15.

2. Locate the point of the vertical line (instrument reading) where it intersects with the horizontal grid line that indicates how much fluoride is present in the sample.

3. Extrapolate the true aluminum concentration by following the curved lines on either side of the intersect point down to the true aluminum concentration.

For example, if the aluminum test result was 0.7 mg/L Al^{3+} and the fluoride present in the sample was 1.0 mg/L F^-, the point where the 0.7 grid line intersects with the 1.0 mg/L F^- grid line falls between the 1.2 and 1.3 mg/L Al curves. In this case, the true aluminum content would be 1.27 mg/L.

Fluoride Interference Graph



Summary of Method

Aluminon indicator combines with aluminum in the sample to form a red-orange color. The intensity of color is proportional to the aluminum concentration. Ascorbic acid is added to remove iron interference. The AluVer 3 Aluminum Reagent, packaged in powder form shows exceptional stability and is applicable for fresh water samples.

REQUIRED REAGENTS

	Cat. No.
Aluminum Reagent Set (100 Tests)	22420-00
Includes: (1) 14290-99, (1) 14577-99, (1) 14294-49	

Description	Quantity Required Per Test	Unit	Cat. No.
AluVer 3 Aluminum Reagent Powder Pillow	1 pillow	100/pkg	14290-99
Ascorbic Acid Powder Pillow	1 pillow	100/pkg	14577-99
Bleaching 3 Reagent Powder Pillow	1 pillow	100/pkg	14294-49

REQUIRED APPARATUS

Cylinders, graduated mixing, 50 mL	1	each	1896-41
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Aluminum Standard Solution, 100 mg/L	100 mL	14174-42
Aluminum Standard Solution, Voluette ampule, 50 mg/L as Al, 10 mL	16/pkg	14792-10
Hydrochloric Acid Solution, 6N (1:1)	500 mL	884-49
m-Nitrophenol Indicator Solution, 10 g/L	100 mL	2476-32
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB	2450-26
Sulfuric Acid Standard Solution, 5.25 N	100 mL MDB	2449-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Brush	each	690-00
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 250 mL	each	14574-46
Fluoride Combination Electrode	each	51928-00
Fluoride ISA Powder Pillows	25/pkg	2589-99
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH/ISE Meter, *sens**ion**™2*, portable	each	51725-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, Volumetric, Class A, 1.00 mL	each	14515-35
Thermometer, –20 to 110 °C, non-mercury	each	26357-02

For Technical Assistance, Price and Ordering

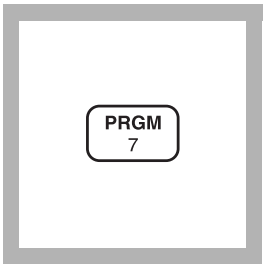
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

BENZOTRIAZOLE (0 to16.0 mg/L) or TOLYLTRIAZOLE (0 to 16.0 mg/L)

UV Photolysis Method*

For cooling or boiler water



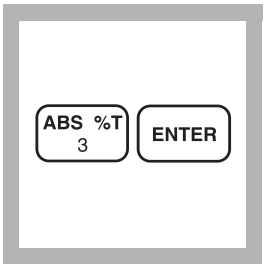
1. Enter the stored program number for benzotriazole (Benzo) or tolyltriazole (Toly).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



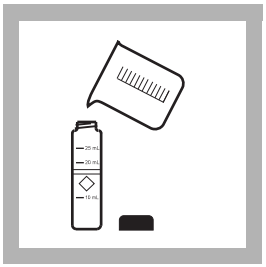
2. Press: **3 ENTER** for either triazole test.

The display will show **mg/L, BENZO,** and the **ZERO** icon

or

the display will show **mg/L, TOLY,** and the **ZERO** icon.

Press the **CONC** key to choose the desired triazole.



3. Fill a sample cell with 25 mL of sample.

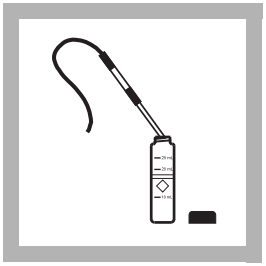
Note: *Sample temperature should be between 20-25 °C (68-77 °F).*

Note: *If sample contains nitrite or borax (sodium borate), adjust the pH to between 4 and 6 with 1 N sulfuric acid.*

4. Add the contents of one Triazole Reagent Powder Pillow. Swirl to dissolve completely.

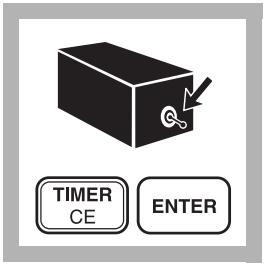
Note: *If the sample contains more than 500 mg/L hardness (as $CaCO_3$), add 10 drops of Rochelle Salt Solution.*

* Adapted from Harp, D., Proceedings 45th International Water Conference, 299 (October 22-24, 1984)



5. Insert the ultraviolet lamp into the sample cell.

Note: *UV safety goggles should be worn while the lamp is on.*

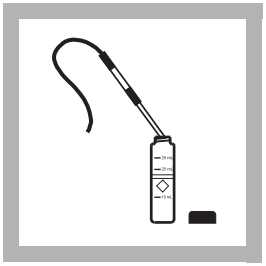


6. Turn the UV lamp ON and press:

TIMER ENTER

A five-minute reaction period will begin.

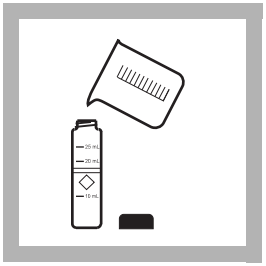
Note: *A yellow color will form if triazole is present.*



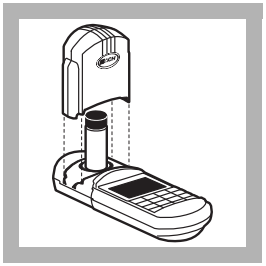
7. When the timer beeps, turn the lamp off and remove it from the cell (the prepared sample). Swirl the cell to mix thoroughly.

Note: *Low results will occur if photolysis (lamp ON) takes place for more or less than five minutes.*

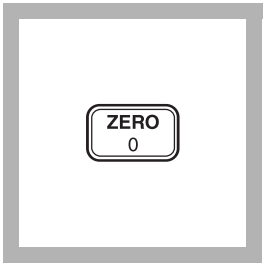
Note: *Avoid handling the quartz surface of the lamp. Rinse the lamp and wipe with a soft, clean tissue between tests.*



8. Fill another sample cell with 25 mL of sample (the blank).



9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **ZERO**

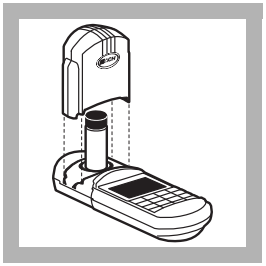
The cursor will move to the right, then the display will show:

0.0 mg/L Benzo

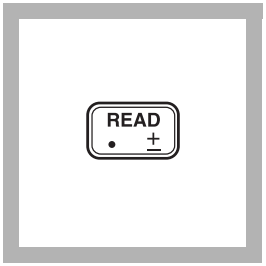
or

0.0 mg/L Toly

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L benzotriazole or tolyltriazole will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling And Storage

The most reliable results are obtained when samples are analyzed as soon as possible after collection.

Accuracy Check

Standard Additions Method

a) Use the TenSette pipet to add 0.1, 0.2 and 0.3 mL of 500-mg/L Benzotriazole Standard Solution to three 25-mL samples. Perform the test according to the above procedure.

Note: *The test will not distinguish between benzotriazole and tolyltriazole.*

b) Each addition of 0.1 mL of standard solution should increase the benzotriazole reading by 2 mg/L over the reading of an unspiked sample.

c) If these increases are not obtained see *Standard Additions* in *Section 1* for more information.

UV Lamp Check

To verify the ultraviolet lamp (normal life equals 5000 hours) is working properly, perform the following test:

a) Prepare a 5.0 mg/L benzotriazole standard solution by pipetting 10.0 mL of Benzotriazole Standard Solution, 500 mg/L benzotriazole, into a 1000-mL volumetric flask. Dilute to volume.

b) Analyze according to the above procedure. If the result is significantly below 5.0 mg/L, replace the lamp.

Method Performance

Precision

In a single laboratory using a standard solution of 9.0 mg/L triazole and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.21 mg/L benzotriazole and ±0.20 mg/L tolyltriazole.

Estimated Detection Limit

The estimated detection limit for program 3 is 0.7 mg/L benzotriazole or tolyltriazole. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Acrylates (as methyl acrylate)	50 mg/L
Alum	400 mg/L
Borate (as sodium tetraborate)	4000 mg/L
Chlorine (as Cl_2)	20 mg/L
Chromium (as chromate)	12 mg/L
Copper	10 mg/L
Hardness	500 mg/L as $CaCO_3$
Iron	20 mg/L
Lignosulfonates	40 mg/L
Magnesium	300 mg/L as $CaCO_3$
Molybdenum (as molybdate)	200 mg/L
Nitrite	4000 mg/L
Phosphonates (AMP or HEDP)	100 mg/L
Sulfate	200 mg/L
Zinc	80 mg/L

Strong oxidizing or reducing agents present in the sample will interfere directly with the test.

Summary of Method

Benzotriazole or tolyltriazole, used in many applications as corrosion inhibitors for copper and copper alloys, are determined by a proprietary catalytic ultraviolet (UV) photolysis procedure requiring less than 10 minutes to perform.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Triazole Reagent Powder Pillows	1 pillow	100/pkg	21412-99

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
Select one based on available voltage:			
Lamp, UV, with power supply, 115 V, 60 Hz, with goggles	1	each	20828-00
Lamp, UV, with power supply, 230 V, 50 Hz, with goggles	1	each	20828-02

OPTIONAL REAGENTS

Benzotriazole Standard Solution, 500 mg/L		100 mL	21413-42
Rochelle Salt Solution		29 mL* DB	1725-33
Sulfuric Acid Standard Solution, 1.00 N		100 mL MDB	1270-32
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Flask, volumetric, Class A, 1000 mL		each	14574-53
Lamp, UV (lamp only)		each	26710-00
pH Paper, 1 to 11 pH		5 rolls/pkg	391-33
pH Meter, *sens**ion***™***1***, portable with electrode		each	51700-10
Pipet Filler, safety bulb		each	14651-00
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
Pipet, volumetric, 10.0 mL, Class A		each	14515-38
Safety Goggles, UV		each	21134-00
Stopwatch		each	14645-00
Thermometer, –20 to 110 °C, non-mercury		each	26357-02
Timer, interval, 1 second to 99 hours		each	23480-00

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

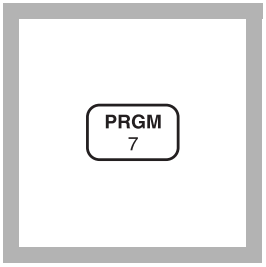
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes.

BROMINE (0 to 4.50 mg/L)

DPD Method* (Powder Pillows or AccuVac Ampuls)

Using Powder Pillows



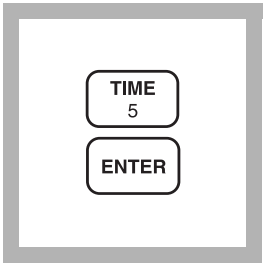
1. Enter the stored program number for bromine (Br_2)-powder pillows.

Press: **PRGM**

The display will show:

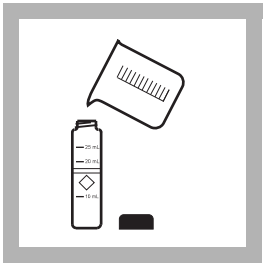
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



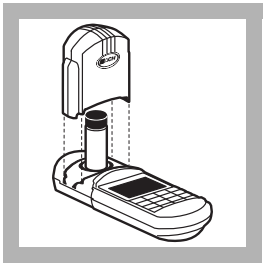
2. Press: **5 ENTER**

The display will show **mg/L, Br2** and the **ZERO** icon**.**



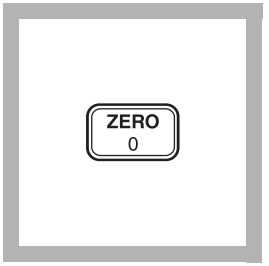
3. Fill a sample cell with 10 mL of sample (the blank).

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

* Adapted from *Standard Methods for the Examination of Water and Wastewater*

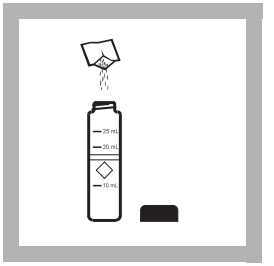


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

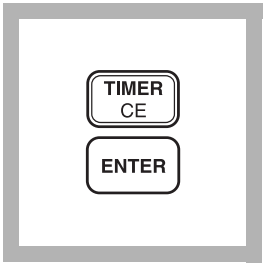
0.00 mg/L Br2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



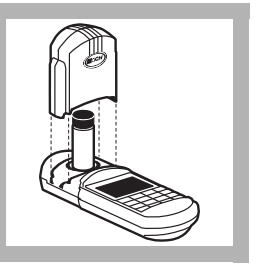
6. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl vigorously to dissolve the powder.

Note: *It is not necessary that all the powder dissolves. A pink color will develop if bromine is present.*

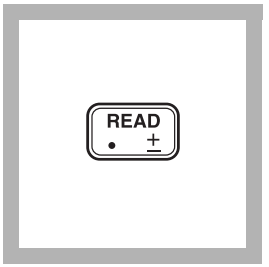


7. Press:**TIMER ENTER**

A three-minute reaction period will begin.



8. When the timer beeps, place the sample into the cell holder. Tightly cover the sample cell with the instrument cap.



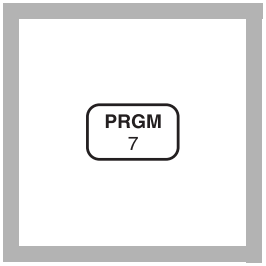
9. Press: **READ**

The cursor will move to the right, then the result in mg/L bromine will be displayed.

Note: *If samples temporarily turn yellow after reagent addition, or the display flashes "limit", it is due to high bromine levels. Dilute fresh samples and repeat the test. A slight loss of bromine may occur during dilution. Multiply results by the dilution factor; see Section 1.*

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Using AccuVac Ampuls



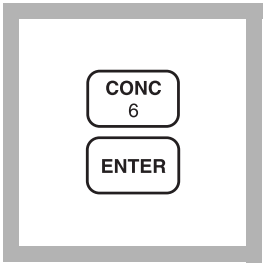
1. Enter the stored program number for bromine (Br_2) AccuVac Ampuls.

Press: **PRGM**

The display will show:

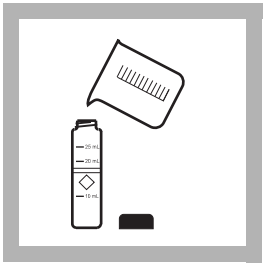
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



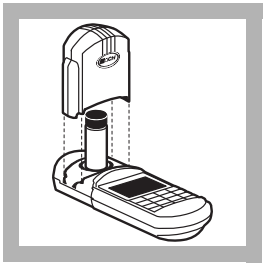
2. Press: **6 ENTER**

The display will show **mg/L, Br2** and the **ZERO** icon**.**

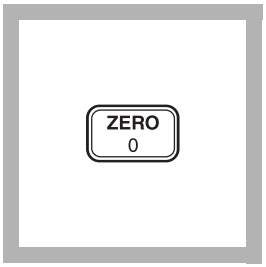


3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



4. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

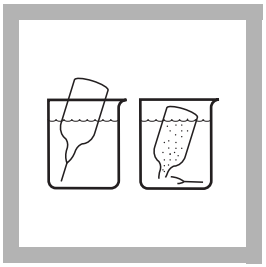


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

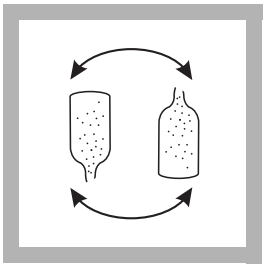
0.00 mg/L Br2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



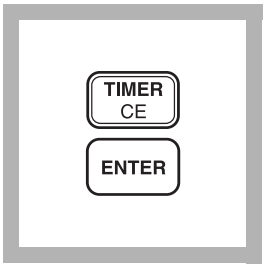
6. Fill one DPD Total Chlorine Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*



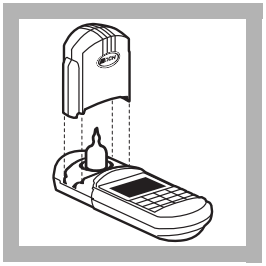
7. Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will form if bromine is present.*

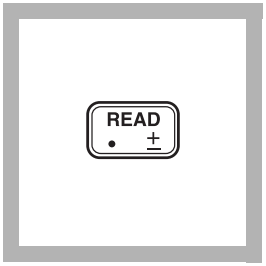


8. Press: **TIMER ENTER**

A three-minute reaction period will begin.



9. After the timer beeps, place the AccuVac ampul into the cell holder. Tightly cover the ampule with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L bromine will be displayed.

Note: *If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high bromine levels. Dilute a fresh sample and repeat the test. A slight loss of bromine may occur during dilution. Multiply the result by the dilution factor; see Section 1.*

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Analyze samples for bromine **immediately** after collection.

Avoid plastic containers since these may have a large bromine demand. **Pretreat glass** sample containers to remove any bromine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for bromine is introduced when a representative sample is not obtained. If sampling from a tap, let the sample flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark.

Perform the bromine analysis immediately after collection.

Accuracy Check

Standard Additions Method (using powder pillows)

a) Snap the top off a LR Chlorine PourRite® Ampule Standard Solution.

b) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.

c) Re-zero the instrument using the original sample (the blank).

d) Place the spiked sample in the cell holder and press **READ**. Record the result.

e) Calculate the equivalent concentration of mg/L bromine added to the sample:

$$\text{mg/L Bromine added} = \frac{0.1\ (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)} \times 2.25}{10.1\ (\text{sample + standard volume})}$$

f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L Br_2 added (step e).

g) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method (using AccuVac Ampuls)

a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.

b) Use a graduated cylinder to measure 25 mL of sample into each of two beakers.

c) Use a TenSette Pipet to add 0.2 mL of the standard to one of the beakers (this is the spiked sample). Swirl to mix.

d) Fill a DPD Total Chlorine AccuVac completely from each beaker.

e) Analyze the spiked and unspiked sample as described in the procedure.

f) Calculate the equivalent concentration of mg/L bromine added to the sample:

$$\text{mg/L Bromine added} = \frac{0.2\ (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)} \times 2.25}{25.2\ (\text{sample + standard volume})}$$

g) The spiked sample result should reflect the analyzed sample result + the calculated mg/L Br_2 added (step f).

h) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 2.34 mg/L bromine and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.02 mg/L bromine.

In a single laboratory using a standard solution of 2.31 mg/L bromine and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation ± 0.02 mg/L bromine.

Estimated Detection Limit

The estimated detection limit for program 5 is 0.04 mg/L Br_2 and 0.03 mg/L Br_2 for program 6. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Chlorine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium , Oxidized (Cr^{6+})	**1.** Adjust sample pH to 6-7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait 1 minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct bromine concentration.
Monochloramine	Interferes at all levels
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See *Interferences, Section 1*.

Summary of Method

Bromine reacts with DPD (N,N-diethyl-p-phenylenediamine) to form a magenta color which is proportional to the total bromine concentration.

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interference will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS (USING POWDER PILLOWS)

Description	Quantity Required Per Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows	1 pillow	100/pkg	21056-69

REQUIRED REAGENTS (USING ACCUVAC AMPULS)

DPD Total Chlorine Reagent AccuVac Ampuls	1 ampule	25/pkg	25030-25

REQUIRED APPARATUS (USING POWDER PILLOWS)

Sample Cells, 10-20-25-mL, w/ cap		6/pkg	24019-06

REQUIRED APPARATUS (USING ACCUVAC AMPULS)

Beaker, 50 mL	1	each	500-41

OPTIONAL REAGENTS

Chlorine Standard Solution, PourRite ampule, 25-30 mg/L, 2 mL		20/pkg	26300-20
DPD Total Chlorine Reagent, SwifTest		250 Tests	28024-00
Potassium Iodide Solution, 30 g/L		100 mL* MDB	343-32
Sodium Arsenite, 5 g/L		100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1.000 N		100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1 N		100 mL* MDB	1270-32
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

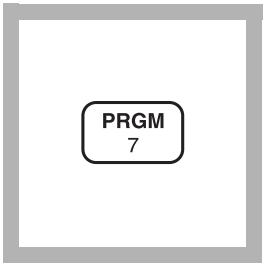
AccuVac Snapper Kit		each	24052-00
PourRite Ampule Breaker		each	24846-00
Cylinder, graduated, 25 mL		each	508-40
pH Meter, *sens**ion***™*1*, portable		each	51700-00
pH Indicator Paper, 1 to 11 pH units		5 rolls/pkg	391-33
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

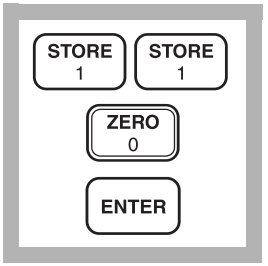
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes



1. Enter the user program number for monochloramine.

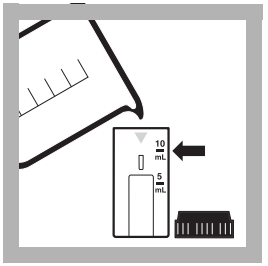
Press: **PRGM**
The display will show:
PRGM?



2. Press:
110 ENTER

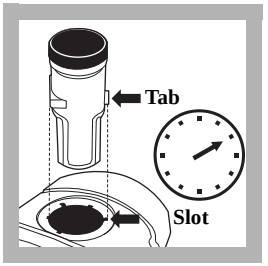
The display will show
mg/L Cl_2
then: **ZERO**

Note: *For alternate forms, press the* **CONC** *key.*



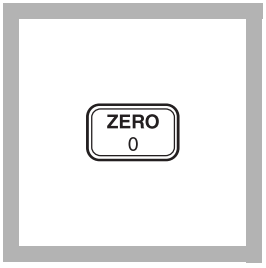
3. Fill the 10-mL/1-cm cell to the 10-mL line with sample.

Note: *For the most accurate results, determine a reagent blank for each new lot of reagent by running the test using deionized water instead of sample.*



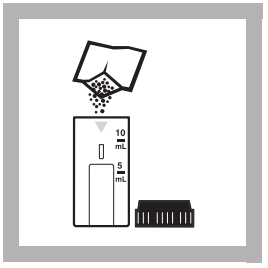
4. Place the cell into the instrument. Tightly cover the sample cell with the instrument cap.

Note: *Place the cell into the cell holder as illustrated. The cell's tab should be at the 2 o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.*

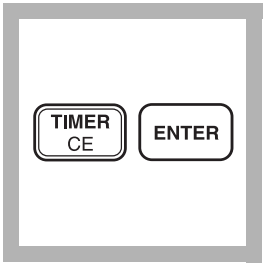


5. Press: **ZERO**

The cursor will move to the right, then the display will show:
0.00 mg/L Cl_2



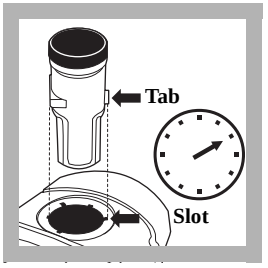
6. Remove the cell from the cell holder and add the contents of one pillow of Monochlor–F to the sample. Cap and shake the cell about 20 seconds to dissolve.



7. Press:
TIMER ENTER

A 5-minute reaction period will begin.

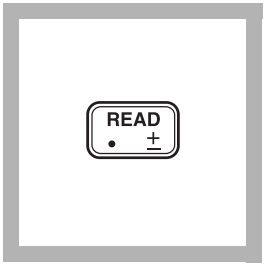
Note: *The color development time depends on the sample temperature. Refer to Table 3 for the actual time required.*



8. After the timer beeps, place the cell into the instrument. Tightly cover the sample cell with the instrument cap.

Note: *Place the cell into the cell holder as illustrated. The cell's tab should be at the 2-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.*

* Patent pending



9. Press: **READ**

The cursor will move to the right, then the result in mg/L monochloramine (as Cl_2 or chosen units) will be displayed.

Sampling and Storage

Analyze samples for monochloramine immediately after collection. If sampling with the sample cell, rinse the sample cell several times with the sample, then carefully fill to the 10-mL mark. If sampling from a tap, let the water flow for at least 5 minutes. Let the container overflow with the sample several times, then cap the container so there is no headspace (air) above the sample.

Accuracy Check

1. Prepare the following monochloramine standard fresh before use.

2. Add the contents of one Buffer Powder Pillow, pH 8.3 to about 50-mL of organic-free water in a clean 100-mL Class A volumetric flask. Swirl to dissolve the powder.

3. Using a Class A volumetric pipet, transfer 2.00 mL of Nitrogen, Ammonia Standard Solution, 100 mg/L as NH_3–N into the flask.

4. Dilute to volume with organic-free water, cap and mix thoroughly. This is a 2.00 mg/L buffered ammonia standard.

5. Pipet 50.00 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a stir bar.

6. Obtain a recent lot of Chlorine Solution Ampules, 50–70 mg/L, and note the actual free chlorine concentration for this lot.

7. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

8. Open an ampule and, using a glass Mohr pipet, add the calculated amount of Chlorine Solution slowly to the ammonia standard, while mixing at medium speed on a stir-plate.

9. Allow the monochloramine solution to mix for 1 minute after all Chlorine Solution is added.

10. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water, cap, and mix thoroughly. This is a nominal 4.5 mg/L (as Cl_2) monochloramine standard.

Use this standard within 1 hour of preparation.

Method Performance

Precision

In a single laboratory, using a monochloramine standard solution of 2.10 mg/L Cl_2 and representative lots of reagent, a single operator obtained a standard deviation of ±0.12 mg/L Cl_2.

Estimated Detection Limit

The estimated detection limit for Method 10171 is 0.05 mg/L Cl_2. For more information on the estimated detection limit, see *Section 1* of the *Procedure Manual*.

Interferences

The following have been tested for interference and found *not* to interfere up to the indicated levels:

Table 9 Non-interfering Substances

Substance	Maximum Level Tested
Alanine	1 mg/L N
Aluminum	10 mg/L
Bromide	100 mg/L Br^-

Table 9 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Bromine	15 mg/L Br_2
Calcium	1000 mg/L $CaCO_3$
Chloride	18,000 mg/L
Chlorine Dioxide	5 mg/L ClO_2
Chromium (III)	5 mg/L
Copper	10 mg/L
Cyanide	10 mg/L CN^-
Free chlorine	10 mg/L Cl_2
Glycine	1 mg/L N
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Lead	10 mg/L
Nitrate	100 mg/L as N
Nitrite	50 mg/L N
Phosphate	100 mg/L PO_4^{3-}
Silica	100 mg/L SiO_2
Silver	10 mg/L
Sulfate	2600 mg/L
Sulfite	50 mg/L SO_3^{2-}
Tyrosine	1 mg/L N
Urea	10 mg/L N
Zinc	5 mg/L

Table 10 Interfering Substances

Interfering Substance and its effect		Interference Level	Recommended Treatment
Magnesium	+	Above 400 mg/L $CaCO_3$	Add 5 drops Rochelle Salt Solution prior to testing.
Manganese (+7)	–	Above 3 mg/L	
Ozone	–	Above 1 mg/L	Usually doesn't coexist with monochloramine.
Sulfide	+	Turns a "rust" color if present.	Usually doesn't coexist with monochloramine.
Thiocyanate	–	Above 0.5 mg/L	

Summary of Method

In the presence of a cyanoferrate catalyst, monochloramine in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample.

Sample Temperature		Minutes
° C	° F	
5	40	10
7	42	9
9	48	8
10	50	8
12	54	7
14	58	7
16	61	6
18	68	4
20	73	3
23	75	2.5
25	77	2
>25	>77	2

Instrument Setup

This procedure will add the current method as a new Hach program to your DR/850 or DR/890.

1. Turn on the instrument by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time, you may use the arrow keys to scroll back to review or change a number already entered.

Line Number	Entry	Line Number	Entry
1	110	29	108
2	42	30	78
3	74	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	63
8	0	36	57
9	0	37	199
10	0	38	104
11	0	39	62
12	64	40	74
13	176	41	61
14	120	42	45
15	106	43	1
16	0	44	204
17	0	45	0
18	0	46	5
19	0	47	10
20	67	48	1
21	108	49	44
22	50	50	0
23	0	51	0
24	0	52	0
25	78	53	0
26	72	54	3
27	50	55	0
28	67	56	255

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Monochlor F Reagent Pillows	1	50/pkg	28022-46

REQUIRED APPARATUS

Sample Cell, 10-mL/1-cm	1	2/pkg	48643-02
Clippers, shears	1	each	23694-00

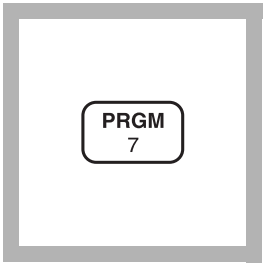
OPTIONAL REAGENTS

Rochelle Salt Solution		29-mL DB	1725-33
Organic-Free Water		500-mL	26415-49
Buffer Powder Pillows, pH 8.3		25/pkg	898-68
Nitrogen, Ammonia Standard Solution, 100 mg/L as NH_3-N		500-mL	24065-49
Chlorine Solution Voluette Ampule, 50–75 mg/L		16/pkg	14268-10

OPTIONAL APPARATUS

Beaker, 100-mL		each	500-42H
Flask, Volumetric, Class A, 100-mL		each	14574-42
Pipet, Mohr, Glass, 10-mL		each	20934-38
Pipet, Volumetric, Class A, 2.00 mL		each	14515-36
Pipet, Volumetric, Class A, 50.00 mL		each	14515-41
Stir Bar, Octagonal		each	20953-52
Stirrer, Magnetic, 110 V, 4” x 4”		each	28812-00

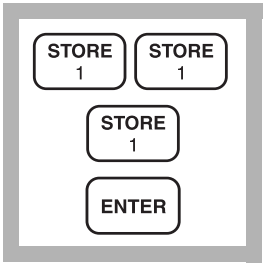
Indophenol Method* **For chlorinated drinking water and chlorinated wastewater**



1. Enter the user program number for Chloramine, HR.

Press: **PRGM**
The display will show:
PRGM?

Note: *For most accurate results, perform a Reagent Blank Correction (Section 1 of the DR/800 Instrument Manual).*



2. Press:
111 ENTER

The display will show:
mg/L Cl_2

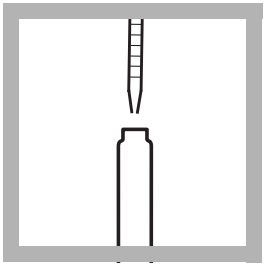
and then

Zero

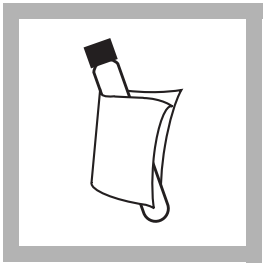
Note: *For alternate forms, press the* **CONC** *key.*

3. Insert the COD/TNT Vial Adapter into the cell holder by rotating the adapter until it drops in place. Push down to fully insert it.

Note: *For better performance, adiffuser band covers the light path holes on the adapter. Do not remove the band.*

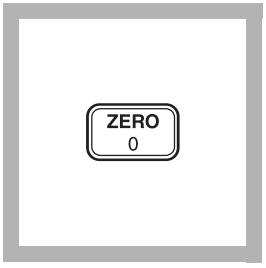


4. Remove the cap from one HR Monochloramine Diluent vial. Use a glass pipet to add 2.0 mL of sample to the vial. Re-cap and invert several times to mix.



5. Wipe the outside of the vial clean.

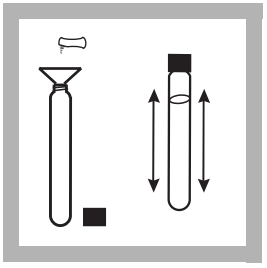
Place the vial into the adapter. Cover the sample vial tightly with the instrument cap.



6. Press: **ZERO**

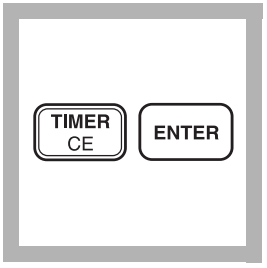
The cursor will move to the right and the display will show:

0.0 mg/L Cl_2.



7. Remove the vial from the cell holder, uncap, and add the contents of one Monochlor–F pillow to the sample. Cap and shake the vial about 20 seconds to dissolve.

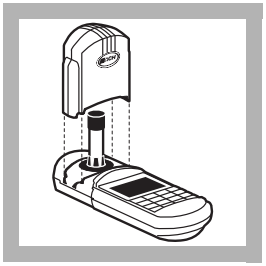
Note: *Use the microfunnel as an aid in adding reagent powder to the vial.*



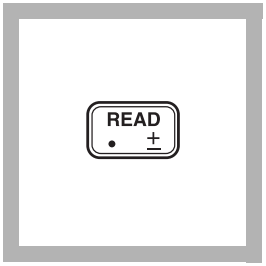
8. Press:
TIMER ENTER

A five-minute reaction period will begin.

* U.S. Patent 6,315,950



9. After the timer beeps, wipe the prepared vial and place it into the instrument. Cover the sample vial tightly with the instrument cap.



10. Press: **READ**.

The cursor will move to the right , then the results in mg/L monochloramine (as Cl_2) will be displayed.

Sampling and Storage

Analyze samples for monochloramine immediately after collection. Rinse the sample container several times with the sample water allowing it to overflow each time. If sampling from a tap, let the water flow for at least 5 minutes. Cap the container so that there is no head space (air) above the sample.

Accuracy Check

Prepare the following monochloramine standard fresh before use:

1. Using a clean 100-mL Class A volumetric flask, add the contents of one Buffer Powder Pillow, pH 8.3, to approximately 50 mL of organic-free water. Swirl to dissolve the powder.

2. Use a Class A volumetric pipet to transfer 2.00 mL of Nitrogen Ammonia Standard Solution, 100-mg/L as NH_3–N, into a flask.

3. Dilute to volume with organic-free water. Cap and mix thoroughly. This is the 2.00-mg/L buffered ammonia standard.

4. Pipet 50.00 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a magnetic stir bar and place the beaker on a stir plate.

5. Note the free chlorine concentration for the Chlorine Solution Ampules, 50–70 mg/L. Use ampules from a recent lot.

6. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

7. Turn the stir plate on to medium speed.

8. Open an ampule. Use a glass Mohr pipet to add the calculated amount of Chlorine Solution slowly to the ammonia standard while it is mixing.

9. Allow the monochloramine solution to mix for 1 minute after all the Chlorine Solution is added.

10. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water. Cap and mix thoroughly. This is a nominal 4.5-mg/L (as Cl_2) monochloramine standard.

Use this solution within 1 hour of preparation.

Method Performance

Precision

In a single laboratory, using a standard solution of 3.5 mg/L monochloramine as chlorine and two representative lots of reagent, a single operator obtained a standard deviation of ± 0.2 mg/L Cl_2.

Estimated Detection Limit

The estimated detection limit (EDL) for Method 10172 is 0.2 mg/L Cl_2. For more information on the EDL, see *Section 1* of the DR/800 Procedure Manual.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 11 Non-interfering Substances

Substance	Maximum Level Tested
Alanine	1 mg/L N
Aluminum	10 mg/L
Bromide	100 mg/L Br^-
Bromine	15 mg/L Br_2
Calcium	1000 mg/L as $CaCO_3$

Table 11 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Chloride	18,000 mg/L
Chlorine Dioxide	5 mg/L ClO_2
Chromium (III)	5 mg/L
Copper	10 mg/L
Cyanide	10 mg/L CN^-
Free Chlorine	10 mg/L Cl_2
Glycine	1 mg/L N
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	1000 mg/L as $CaCO_3$
Manganese (VII)	10 mg/L
Lead	10 mg/L
Nitrate	100 mg/L N
Nitrite	50 mg/L N
Phosphate	100 mg/L PO_4
Silica	100 mg/L SiO_2
Silver	10 mg/L
Sulfate	2600 mg/L
Sulfite	50 mg/L SO_3^{2-}
Tyrosine	1 mg/L as N
Urea	10 mg/L as N
Zinc	5 mg/L

Table 12 Interfering Substances

Interfering Substance and its effect		Interference Level	Recommended Treatment
Ozone	–	Above 1 mg/L	Usually doesn't coexist with monochloramine
Sulfide	+	Turns a "rust" color if present.	Usually doesn't coexist with monochloramine
Thiocyanate	–	Above 0.5 mg/L	

Summary of Method

The sample is first diluted in a Test 'N Tube™. In the presence of a cyanoferrate catalyst, monochloramine (NH_2Cl) in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate compound couples with excess substituted phenol to form a green indophenol. Color intensity is proportional to the amount of monochloramine present in the sample.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet (MSDS) for information specific to the reagent used.

Instrument Setup

This procedure will add the current method as a new Hach program to your DR/850 or DR/890 instrument.

1. Turn the instrument on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Key in "8138", then press **ENTER**.
6. Key the number in the "Enter" column corresponding to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

Table 13

Line number on display	Enter	Line number on display	Enter
1	111	29	108
2	42	30	78
3	73	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	63
8	0	36	58
9	0	37	61
10	0	38	112

Table 13 (Continued)

11	0	39	62
12	65	40	74
13	116	41	61
14	49	42	112
15	248	43	0
16	0	44	110
17	0	45	0
18	0	46	0
19	0	47	10
20	67	48	1
21	108	49	44
22	50	50	0
23	0	51	0
24	0	52	0
25	78	53	0
26	72	54	153
27	50	55	0
28	67	56	255

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
HR Monochloramine Test 'N Tubes, 50 tests			28051-45
Includes:			
HR Monochloramine Diluent Vials	50		*
Funnel, micro	1	each	25843-35
Monochlor F Reagent Pillows	1	50/pkg	28022-46

REQUIRED APPARATUS

COD/TNT Vial Adapter, DR/800	1	each	48464-00
Pipet, Mohr, glass, 2.00-mL	1	each	20936-36
Test Tube Rack	1	each	18641-00

OPTIONAL REAGENTS

Organic-free Water	500-mL		26415-49
Buffer Powder Pillows, pH 8.3	25/pkg		898-68
Nitrogen, Ammonia Standard Solution, 100-mg/L as NH_3–N	500-mL		24065-49
Chlorine Solution Voluette® Ampule, 50–75 mg/L, 10-mL	16/pkg		14268-10

OPTIONAL APPARATUS

Beaker, 100-mL		each	500-42H
Clippers (medium powder pillows)		each	968-00
Clippers (shears)		each	23694-00
Flask, Volumetric, Class A, 100-mL		each	14574-42
Pipet, Mohr, Glass, 10-mL		each	20934-38
Pipet, Volumetric, Class A, 2.00-mL		each	14515-36
Pipet, Volumetric, Class A, 50.00-mL		each	14515-41
Stir Bar, Octagonal		each	20953-52
Stirrer, Magnetic, 110 V, 4" x 4"		each	23436-00

* Not sold separately.

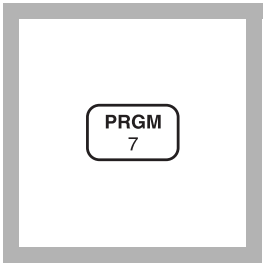
CHLORINE DIOXIDE (0 to 5.00 mg/L) Method 10126

DPD Method* **For water**

USEPA accepted for reporting for drinking water analysis

Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*

Using Powder Pillows

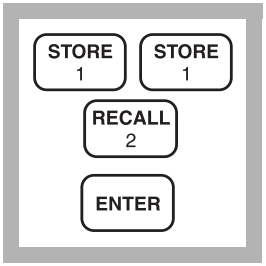


1. Enter the stored program number for chlorine dioxide (ClO_2) powder pillows.

Press: **PRGM**

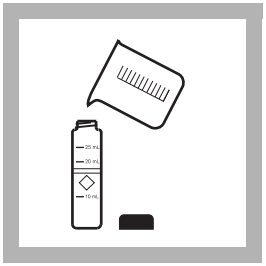
The display will show:

PRGM ?



2. Press: **112 ENTER**

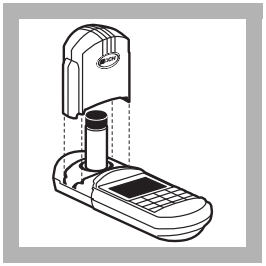
The display will show **mg/L, ClO2**, and the **ZERO** icon.



3. Fill a sample cell with 10 mL of sample (the blank).

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

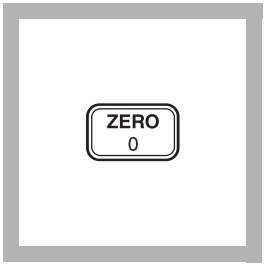
Note: *Wipe off any liquid or fingerprints before inserting the sample cell into the instrument.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *For best results, run a reagent blank using deionized water as the sample. Subtract the blank value from the sample reading to obtain the final result. See* Reagent Blank Correction *in Section 1 of the DR/800 Procedure Manual.*

* Procedure is equivalent to *Standard Method 4500, $Cl0_2P$*

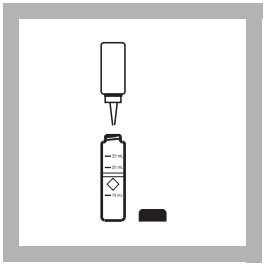


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L ClO2

Note: *If Reagent Blank Correction is on, the display may flash* **"limit"**. *See Section 1 of the DR/800 Procedures Manual.*

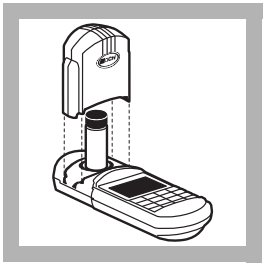


6. Add four drops of Glycine Reagent to the sample cell. Swirl to mix.

7. Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl to mix.

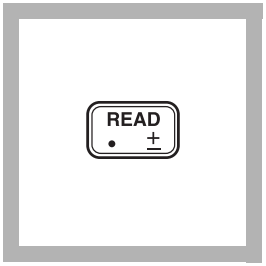
Note: *A pink color will develop if free chlorine dioxide is present.*

Note: *Perform step 9 within one minute of reagent addition.*



8. Allow 30 seconds for undissolved powder to settle. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Wipe off any liquid or fingerprints before inserting the sample cell into the instrument.*

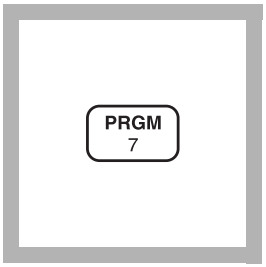


9. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine dioxide will be displayed.

Note: *If the sample temporarily turns yellow after reagent addition, or the display flashes* **"limit"**, *it is due to high chlorine dioxide levels. Dilute a fresh sample with chlorine dioxide-free water and repeat the test. A slight loss of chlorine dioxide may occur during dilution. Multiply the result by the dilution factor.*

Using AccuVac® Ampuls

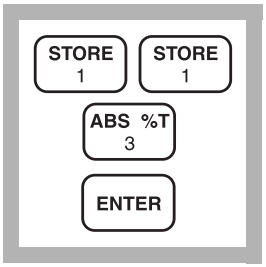


1. Enter the stored program number for chlorine dioxide (ClO_2) AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?



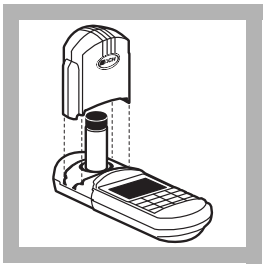
2. Press: **113 ENTER**

The display will show **mg/L, ClO2** and the **ZERO** icon.

3. Fill a sample cell with at least 10 mL of sample (the blank). Fill a 50-mL beaker with 40 mL of sample. Using the correct sample volume is important.

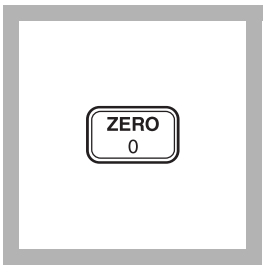
Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

Note: *Wipe off any liquid or fingerprints before inserting the sample cell into the instrument.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *For best results, run a reagent blank using deionized water as the sample. Subtract the blank value from the sample reading to obtain the final result. See* Reagent Blank Correction *in Section 1 of the DR/800 Procedure Manual.*

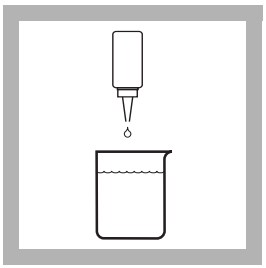


5. Press: **ZERO**

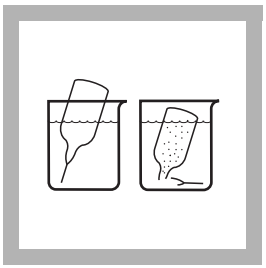
The cursor will move to the right, then the display will show:

0.00 mg/L ClO2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1 of the DR/800 Procedures Manual.*



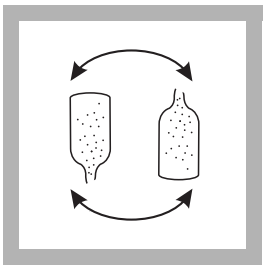
6. Add 16 drops of Glycine Reagent to the sample in the beaker. Swirl to mix.



7. Fill a DPD Free Chlorine Reagent AccuVac Ampul with sample.

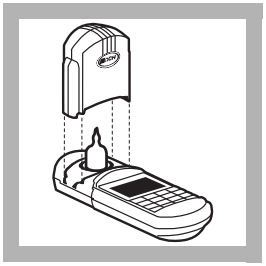
Note: *Keep the tip immersed while the ampul fills completely.*

Note: *Perform step 10 within one minute of reagent addition.*

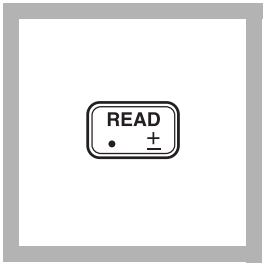


8. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will form if chlorine dioxide is present.*



9. Allow 30 seconds for undissolved powder to settle. Place the AccuVac Ampul into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine dioxide will be displayed.

Note: *If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine dioxide levels. Dilute a fresh sample with chlorine dioxide-free water and repeat the test. A slight loss of chlorine dioxide may occur during dilution. Multiply the result by the dilution factor.*

Sampling and Storage

Analyze samples for chlorine dioxide **immediately** after collection. Chlorine dioxide is a strong oxidizing agent, and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of chlorine dioxide in water.

Avoid plastic containers since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine dioxide demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for chlorine dioxide is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the analysis immediately.

Accuracy Check

Because chlorine dioxide is difficult and hazardous to produce, check the DPD and glycine reagents by using chlorine standards. Proceed as follows:

1. Prepare a 1-mg/L free chlorine standard.

Method 1

a. Obtain Free Chlorine Standards, (Cat. No. 14268-10).

b. Determine the concentration of the standard from the certificate of analysis shipped with the standard (50-75 mg/L). Calculate the volume of standard needed as follows:

mL standard needed = 100 ÷ standard concentration

c. Pipet the volume of standard needed into a 100-mL volumetric flask. Dilute to the line with chlorine demand-free deionized water. Invert to mix.

Method 2

a. Dilute 1 drop of commercial 5% chlorine bleach in 1 liter of chlorine demand-free deionized water. Use this as the standard.

2. Verify the standard's concentration using the Hach Free Chlorine Method, #8021.

3. Perform the chlorine dioxide test on the standard without adding glycine (*step 6*).

4. The chlorine dioxide reading should be about 2.45 times greater than the chlorine result. If so, this verifies the DPD and the instrument are functioning properly.

5. Repeat the chlorine dioxide test on the chlorine standard, including the glycine addition (*step 6*). The reading should be less than 0.10 mg/L. This verifies that the glycine is eliminating free chlorine interference.

Method Performance

Precision

Program	Standard	95% Confidence Limits
112	0.24 mg/L	0.22–0.26 mg/L ClO_2
112	4.79 mg/L	4.67–4.91 mg/L ClO_2
113	0.26 mg/L	0.21–0.27 mg/L ClO_2
113	4.83 mg/L	4.71–4.97 mg/L ClO_2

For more information on determining precision data and method detection limits, see *Section 1* of the *DR/800 Procedures Manual*.

Estimated Detection Limit (EDL)

Program	EDL
112	0.04 mg/L ClO_2
113	0.04 mg/L ClO_2

For more information on derivation and use of Hach's estimated detection limit, see *Section 1* of the *DR/800 Procedures Manual*.

Interferences

A substance interferes if it changes the final reading by 0.1 mg/L ClO_2 or more.

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (see *Section 1, Correction For Volume Additions, in the DR/800 Procedures Manual).*
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6–7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (see *Section 1, Correction For Volume Additions, in the DR/800 Procedures Manual).*
Bromine, Br_2	Interferes at all levels.
Chlorine, Cl_2	May interfere at levels greater than 6 mg/L. Additional glycine may be able to compensate for this interference.
Chloramines, organic	May interfere.
Flocculating agents	High levels of most flocculating agents can be tolerated. This tolerance is decreased if chlorine is present. See the information about metals in this table. In the presence of 0.6 mg/L Cl_2, $Al(SO_4)_3$ (< 500 mg/L) and $FeCl_2$ (<200 mg/L) may be tolerated.
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$.

Interfering Substance	Interference Levels and Treatments
Iodine, I_2	Interferes at all levels.
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	Oxidized manganese interferes at all levels. Oxidized chromium interferes at levels greater than 2 mg/L. To remove the interferences: **1.** Adjust sample pH to 6–7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result of this test from the original analysis to obtain the correct chlorine dioxide concentration.
Metals	Various metals may interfere by combining with the glycine needed to remove the chlorine interference. Metal interference is limited except when chlorine is present. In the presence of 0.6 mg/L Cl_2, both copper (>10 mg/L) and nickel (>50 mg/L) interfere. Other metals may also interfere, depending on their ability to prevent glycine from reacting with any Cl_2 in the sample. It may be necessary to add more glycine to overcome this interference.
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L ClO_2 increase in the reading.
Ozone	Interferes at levels greater than 1.5 mg/L.
Peroxides	May interfere.
Extreme sample pH	Adjust to pH 6–7. See *Section 1, pH Interferences, in the DR/800 Procedures Manual.*
Highly buffered samples	Adjust to pH 6–7. *See Section 1, pH Interferences, in the DR/800 Procedures Manual.*

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004).

Summary of Method

Chlorine dioxide reacts with DPD (N,N-diethyl-p-phenylenediamine) Indicator Reagent (to the extent of one-fifth of its total available chlorine content corresponding to reduction of chlorine dioxide to chlorite) to form a pink color. The color intensity is proportional to the ClO_2 in the sample. Chlorine interference is eliminated by adding glycine, which converts free chlorine to chloroaminoacetic acid, but has no effect on chlorine dioxide at the test pH.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required per test	Unit	Cat. No.
Chlorine Dioxide DPD/Glycine Reagent Set (100 tests)			27709-00
Includes one of each:			
DPD Free Chlorine Reagent Powder Pillows, 10 mL	1 pillow	100/pkg	21055-69
Glycine Reagent	4 drops	29 mL	27621-33

REQUIRED REAGENTS (Using AccuVac® Ampuls)

Chlorine Dioxide DPD/Glycine AccuVac® Ampul Reagent Set (25 tests)			27710-00
Includes one of each:			
DPD Free Chlorine Reagent AccuVac® Ampuls	1	25/pkg	25020-25
Glycine Reagent	16 drops	29 mL	27621-33

OPTIONAL REAGENTS

Chlorine Standard Solution, Voluette™ ampule, 50-75 mg/L, 10 mL		16/pkg	14268-10
DPD Free Chlorine Reagent, SwifTest™		250 tests	28023-00
Potassium Iodide Solution, 30 g/L		100 mL* MDB	343-32
Sodium Arsenite, 5 g/L		100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1.000 N		100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N		100 mL* MDB	1270-32
Water, deionized		4 L	272-56
Water, sterile, chlorine dioxide-free		500 mL	26415-49

OPTIONAL APPARATUS

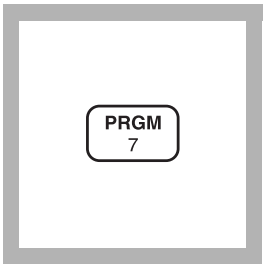
AccuVac® Snapper Kit		each	24052-00
Cylinder, graduated, 25 mL		each	508-40
pH Meter, *sens**ion**™1*, portable, with electrode		each	51700-10
pH Paper, 1 to 11 pH units		5 rolls/pkg	391-33
Pipet, TenSette®, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette® Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette® Pipet		1000/pkg	21856-28
PourRite™ Ampule Breaker		each	24846-00

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Marked Dropper Bottle - contact Hach for larger sizes.

CHLORINE DIOXIDE, Mid Range (0 to 50.0 mg/L) For water and wastewater

Direct Reading Method

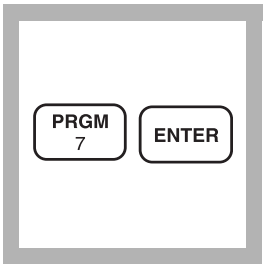


1. Enter the stored program number for mid-range chlorine dioxide (ClO_2).

Press: **PRGM**

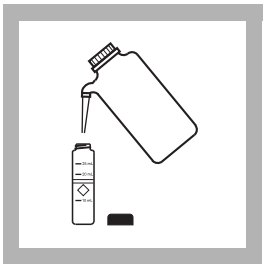
The display will show:

PRGM ?



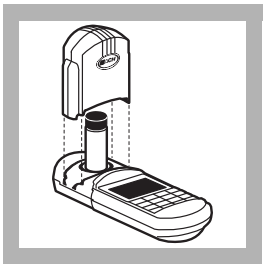
2. Press: **7 ENTER**

The display will show **mg/L, ClO2** and the **ZERO** icon**.**

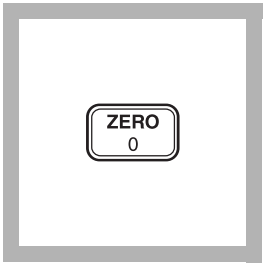


3. Fill a sample cell (the blank) with 10 mL of deionized water.

Note: *Analyze samples immediately after collection.*



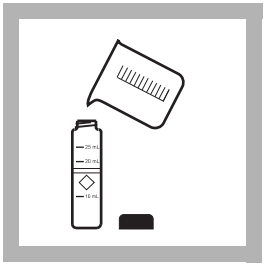
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



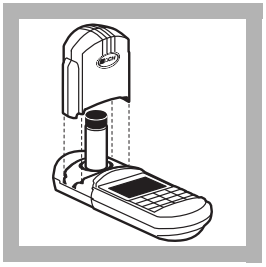
5. Press: **ZERO**

The cursor will move to the right, then the display will show:

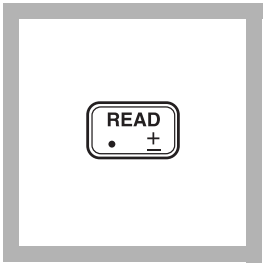
0.0 mg/L ClO2



6. Fill another sample cell with 10 mL of sample (the prepared sample).



7. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine dioxide will be displayed.

Note: *If the display flashes "limit" it is due to high ClO_2 levels. A slight loss of chlorine dioxide may occur during dilution. Dilute a fresh sample and repeat the test. Multiply the result by the dilution factor; see Section 1.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Chlorine dioxide is very volatile and unstable; analyze samples immediately upon collection.

Accuracy Check

Standard Solution Method

Preparing chlorine dioxide standards is difficult and dangerous. In addition, **these standards are both explosive and volatile!** Only a trained chemist should prepare the standards using appropriate safety equipment and precautions. Hach does not recommend independent standard preparation of chlorine dioxide standards. If independent standard preparation is required, please refer to the instructions in *Standard Methods for the Examination of Water and Wastewater*, 19th ed., under the headings "Stock chlorine dioxide solution" and "Standard chlorine dioxide solution" (pg. 4-54).

Method Performance

Precision

In a single laboratory, using a standard solution of 25.0 mg/L ClO_2, a single operator obtained a standard deviation of ±0.3 mg/L ClO_2. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 7 is 7.3 mg/L ClO_2. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

Chlorine dioxide, a yellow gas, can be measured directly in a water solution. This method uses a wavelength of 420 nm to increase the range of the test.

REQUIRED REAGENTS AND APPARATUS

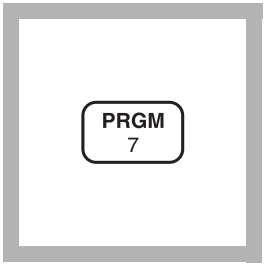
Description	Quantity Required Per Test	Unit	Cat. No.
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06
Water, deionized	10 mL	4 L	272-56

Outside the U.S.A.—Contact the Hach office or distributor serving you.

DPD Method

USEPA accepted for reporting drinking water analyses*
For testing higher levels of free chlorine (hypochlorous acid and hypochlorite) in drinking water, cooling water, and industrial process waters

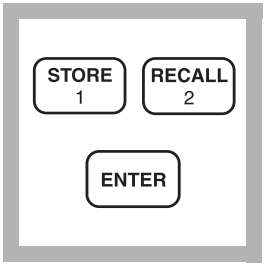
Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



1. Enter the user program number for Chlorine, UHR.

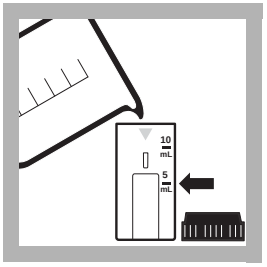
Press: **PRGM**
The display will show:
PRGM?

Note: *If the chlorine is typically less than 2.0 mg/L, use method 8021, program number 9.*

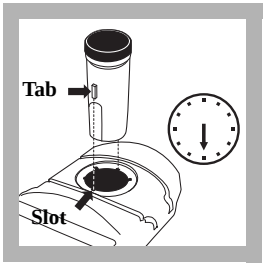


2. Press:
12 ENTER

The display will show
mg/L Cl_2
then: **ZERO**



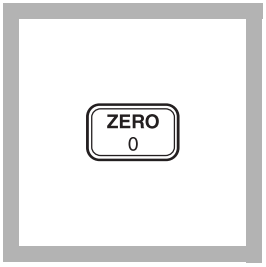
3. Fill the 10-mL/1-cm cell to the 5-mL line with sample.



4. Place the cell into the instrument. Cover the sample cell tightly with the instrument cap.

Note: *Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6 o'clock position and completely seated in the cell holder slot.*

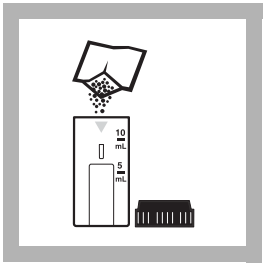
* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-C1-G for drinking water.



5. Press: **ZERO**

The cursor will move to the right, then the display will show:

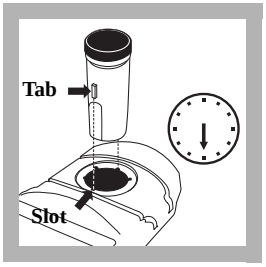
0.0 mg/L Cl_2



6. Remove the sample cell from the cell holder and add the contents of one 25-mL DPD Free Chlorine Reagent pillow to the sample. Cap and shake the sample cell about 20 seconds to dissolve.

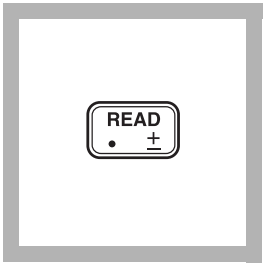
Proceed **immediately** to *step 7*.

Note: *A pink color will develop if chlorine is present.*



7. Place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

Note: *Place the sample cell into the cell holder as illustrated. The sample cell tab should be at the 6-o'clock position and completely seated in the cell holder slot.*



8. Within one minute after reagent addition, press: **READ**.

The cursor will move to the right. The result in mg/L chlorine (as Cl_2) will be displayed.

Note: *See "Interferences" on page 120 for samples with high monochloramine concentrations.*

Sampling and Storage

Analyze samples for chlorine immediately after collection. Free chlorine is a strong oxidizing agent and reacts rapidly with various compounds. Many factors such as sunlight, pH, temperature, and sample composition will influence decomposition of free chlorine in water.

- Avoid plastic containers which may have a large chlorine demand.
- Pretreat glass sample containers to remove chlorine demand by soaking in a dilute bleach solution (1 mL of commercial bleach to 1 liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.
- Use separate, dedicated sample cells for free and total chlorine determinations. If trace iodide from the total chlorine reagent is carried over to the free chlorine test, monochloramine could interfere.

- A common error in testing for chlorine is failure to obtain a representative sample. If sampling from a tap, let the water flow for at least five minutes to ensure a representative sample. Let the sample container overflow with sample several times. Cap the container so there is no air above the sample.
- If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 5-mL mark. Proceed with the chlorine test immediately.

Accuracy Check

1. Fill three mixing cylinders (Cat. No. 20886-38) with 5-mL of sample.
2. Snap the neck of a HR Chlorine Ampule Standard, 50–75 mg/L Cl_2. Using the TenSette® Pipet, add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each cylinder and mix thoroughly.
3. Analyze each standard addition sample as described in the procedure. Record each result.
4. Calculate the concentration of mg/L chlorine added to each sample.

$$\text{mg/L chlorine added} = \frac{\text{volume of standard added} \times \text{label value of } Cl_2 \text{ standard ampule}}{\text{sample volume} + \text{volume of standard added}}$$

The spiked sample results should reflect the analyzed sample result plus the calculated mg/L Cl_2 added to each sample. If these increases do not occur, see Standard Additions in Section 1 of a DR/800 Procedure Manual for more information.

Method Performance

Precision

In a single laboratory, using a chlorine standard solution of 5.05 mg/L Cl_2 and representative lots of reagent, a single operator obtained a standard deviation of ± 0.05 mg/L Cl_2.

Estimated Detection Limit

The estimated detection limit for Method 10069 is 0.1 mg/L Cl_2. For more information on the estimated detection limit, see Section 1 of the DR/800 Procedure Manual.

Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. **1.** Neutralize to pH 6–7 with 1 N Sodium Hydroxide. **2.** Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested. **3.** Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. **1.** Neutralize to pH 6–7 with 1 N Sulfuric Acid. **2.** Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested. **3.** Correct for volume addition.
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Iodine, I_2	Interferes at all levels
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7. **2.** Add 2 drops Potassium Iodide (30 g/L) to a 5-mL sample. **3.** Mix and wait 1 minute. **4.** Add 2 drops of Sodium Arsenite (5 g/L) and mix. **5.** Analyze the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.

<table>
<tr><th>Interfering Substance</th><th>Interference Levels and Treatments</th></tr>
<tr><td>Monochloramine</td><td>For conventional free chlorine disinfection (beyond the breakpoint), monochloramine concentrations are very low. If monochloramine is present in the sample, its interference in the free chlorine test varies with the sample temperature, the relative amount of monochloramine to free chlorine, and the time required to do the analysis. Approximate interference levels of monochloramine in the free chlorine test are listed below (as mg/L Cl_2).
<table>
<tr><th>NH_2Cl</th><th colspan="4">Sample Temperature °C (°F)</th></tr>
<tr><th>(as Cl_2)</th><th>5 (40)</th><th>10 (50)</th><th>20 (68)</th><th>30(83)</th></tr>
<tr><td>1.2</td><td>0.2</td><td>0.2</td><td>0.3</td><td>0.3</td></tr>
<tr><td>2.5</td><td>0.4</td><td>0.5</td><td>0.6</td><td>0.6</td></tr>
<tr><td>3.5</td><td>0.5</td><td>0.6</td><td>0.7</td><td>0.8</td></tr>
</table></td></tr>
<tr><td>Ozone</td><td>Interferes at all levels</td></tr>
<tr><td>Peroxides</td><td>May interfere</td></tr>
<tr><td>Extreme sample pH or highly buffered samples</td><td>Adjust the sample pH to 6–7 with Sulfuric Acid or Sodium Hydroxide</td></tr>
</table>

Summary of Method

The range of analysis using the DPD method for free chlorine can be extended by adding more indicator in proportion to sample volume. Thus, a larger fill powder pillow of DPD Free Chlorine Reagent is added to a 5-mL sample portion.

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) reacts immediately with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color which is proportional in intensity to the chlorine concentration.

Instrument Setup

The following procedure will add this method as a new Hach program to a DR/800 instrument.

1. Turn on the instrument by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the **DOWN** arrow key until the prompt line shows USER.
4. Press the **ENTER** key.
5. Enter “8138”, followed by **ENTER**.

6. Key the number in the "Enter" column corresponding to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

Line Number	Enter	Line Number	Enter
1	12	29	0
2	24	30	0
3	73	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	62	36	0
9	55	37	0
10	23	38	0
11	88	39	0
12	64	40	0
13	113	41	0
14	242	42	0
15	18	43	0
16	0	44	110
17	0	45	0
18	0	46	0
19	0	47	10
20	67	48	0
21	108	49	180
22	50	50	0
23	0	51	0
24	0	52	0
25	0	53	0
26	0	54	236
27	0	55	0
28	0	56	255

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
DPD Free Chlorine Reagent Powder Pillows, 25-mL	1	100/pkg	14070-99

REQUIRED APPARATUS

Sample Cell, 10-mL/1-cm	1	2/pkg	48643-02

OPTIONAL REAGENTS

Chlorine Standard Solution, 2-mL Voluette® Ampule, 50–75 mg/L		20/pkg	14268-20
Potassium Iodide Solution, 30-g/L		100 mL MDB	343-32
Sodium Arsenite Solution, 5-g/L		100 mL MDB	1047-32
Sodium Hydroxide Standard Solution, 1.00 N		100 mL MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N		100 mL MDB	1270-32
Water, deionized		4 L	272-56

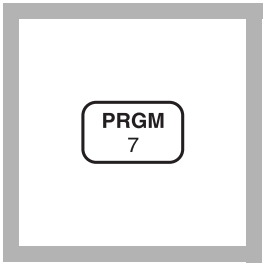
OPTIONAL APPARATUS

Ampule Breaker Kit		each	24846-00
Cylinder, graduated, 10-mL, mixing		each	20886-38
pH Meter, sens***ion***™1, portable, with electrode		each	51700-10
Pipet, TenSette®, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28

CHLORINE, TOTAL, Ultra-High Range (0.0–10.0 mg/L Cl_2) Method 10070

DPD Method **USEPA accepted for reporting water and wastewater analyses***
For testing higher levels of total chlorine (free and combined) in drinking water, cooling water, industrial process waters, or treated wastewater

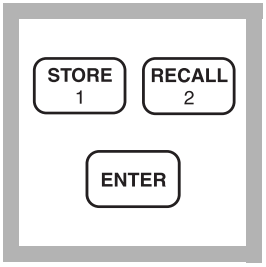
Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



1. Enter the user program number for Chlorine, UHR.

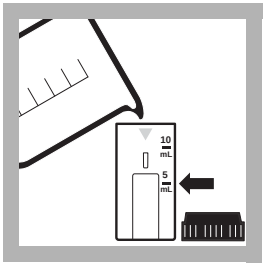
Press: **PRGM**
The display will show:
PRGM?

Note: *If the chlorine is typically less than 2.0 mg/L, use method 8167, program number 9.*

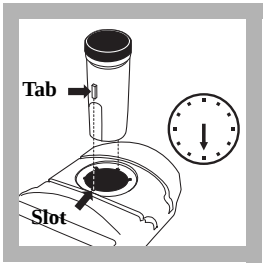


2. Press:
12 ENTER

The display will show
mg/L Cl_2
then: **ZERO**



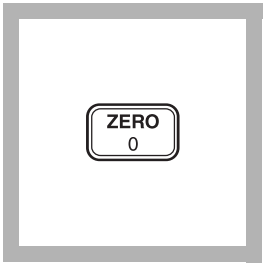
3. Fill the 10-mL/1-cm cell to the 5-mL line with sample.



4. Place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

Note: *Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6 o'clock position and completely seated in the cell holder slot.*

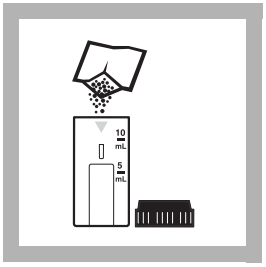
* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-C1-G for drinking water.



5. Press: **ZERO**

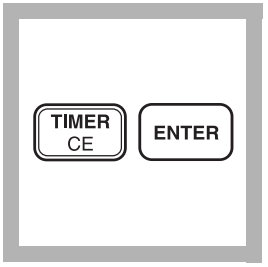
The cursor will move to the right, then the display will show:

0.0 mg/L Cl_2



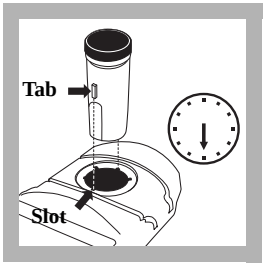
6. Remove the sample cell from the cell holder and add the contents of one 25- mL DPD Total Chlorine Reagent pillow to the sample. Cap and shake the sample cell about 20 seconds to dissolve.

Note: *A pink color will develop if chlorine is present.*



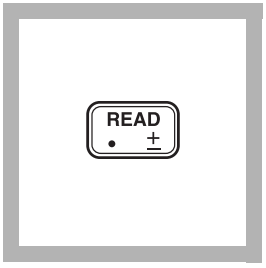
7. Press:
TIMER ENTER

A 3-minute reaction period will begin.



8. Within 3 minutes after the timer beeps, place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

Note: *Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6-o'clock position and completely seated in the cell holder slot.*



9. Press: **READ**

The cursor will move to the right. The result in mg/L chlorine (as Cl_2) will be displayed.

Sampling and Storage

Analyze samples for chlorine immediately after collection. Free and combined chlorine are strong oxidizing agents and react rapidly with various compounds. Many factors such as sunlight, pH, temperature, and sample composition will influence decomposition of chlorine in water.

- Avoid plastic containers which may have a large chlorine demand.
- Pretreat glass sample containers to remove chlorine demand by soaking in a dilute bleach solution (1 mL of commercial bleach to 1 liter of deionized water) for at least one hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.
- Use separate, dedicated sample cells for free and total chlorine determinations. If trace iodide from the total chlorine reagent is carried over to the free chlorine test, monochloramine could interfere.
- A common error in testing for chlorine is failure to obtain a representative sample. If sampling from a tap, let the water flow for at least five minutes to ensure a representative sample. Let the sample container overflow with sample several times. Cap the container so there is no air above the sample.
- If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 5-mL mark. Proceed with the chlorine test immediately.

Accuracy Check

1. Fill three mixing cylinders (Cat. No. 20886-38) with 5-mL of sample.

2. Snap the neck of a HR Chlorine Ampule Standard, 50–75 mg/L Cl_2. Using the TenSette® Pipet, add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each cylinder and mix thoroughly.

3. Analyze each standard addition sample as described in the procedure. Record each result.

4. Calculate the concentration of mg/L chlorine added to each sample.

$$\text{mg/L chlorine added} = \frac{\text{volume of standard added} \times \text{label value of } Cl_2 \text{ standard ampule}}{\text{sample volume + volume of standard added}}$$

The spiked sample results should reflect the analyzed sample result plus the calculated mg/L Cl_2 added to each sample. If these increases do not occur, see Standard Additions in Section 1 of a DR/800 Procedure Manual for more information.

Method Performance

Precision

In a single laboratory, using a chlorine standard solution of 5.05 mg/L Cl_2 and representative lots of reagent, a single operator obtained a standard deviation of ± 0.05 mg/L Cl_2.

Estimated Detection Limit

The estimated detection limit for Method 10070 is 0.05 mg/L Cl_2. For more information on the estimated detection limit, see Section 1 of a DR/800 Procedure Manual.

Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. **1.** Neutralize to pH 6–7 with 1 N Sodium Hydroxide. **2.** Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested. **3.** Correct for volume addition.
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. **1.** Neutralize to pH 6–7 with 1 N Sulfuric Acid. **2.** Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested. **3.** Correct for volume addition.
Bromine, Br_2	Interferes at all levels
Chlorine Dioxide, ClO_2	Interferes at all levels
Chloramines, organic	May interfere
Iodine, I_2	Interferes at all levels

Interfering Substance	Interference Levels and Treatments
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium, oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7. **2.** Add 2 drops Potassium Iodide (30 g/L) to a 5-mL sample. **3.** Mix and wait 1 minute. **4.** Add 2 drops of Sodium Arsenite (5 g/L) and mix. **5.** Analyze the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH or highly buffered samples	Adjust the sample pH to 6–7 with Sulfuric Acid or Sodium Hydroxide

Summary of Method

The range of analysis using the DPD method for total chlorine can be extended by adding more indicator in proportion to sample volume. Thus, a larger fill powder pillow of DPD Total Chlorine Reagent is added to a 5-mL sample portion.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N,N-diethyl-p-phenylenediamine) along with free chlorine present in the sample to form a pink color which is proportional in intensity to the total chlorine concentration.

Instrument Setup

The following procedure will add this method as a new Hach program to a DR/800 instrument.

1. Turn on the instrument by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the **DOWN** arrow key until the prompt line shows USER.
4. Press the **ENTER** key.
5. Enter “8138”, followed by **ENTER**.
6. Key the number in the “Enter” column corresponding to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

Line Number	Enter	Line Number	Enter
1	12	29	0
2	24	30	0
3	73	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	62	36	0
9	55	37	0
10	23	38	0
11	88	39	0
12	64	40	0
13	113	41	0
14	242	42	0
15	18	43	0
16	0	44	110
17	0	45	0
18	0	46	0
19	0	47	10
20	67	48	0
21	108	49	180
22	50	50	0
23	0	51	0
24	0	52	0
25	0	53	0
26	0	54	236
27	0	55	0
28	0	56	255

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows, 25-mL	1	100/pkg	14064-99

REQUIRED APPARATUS

Sample Cell, 10-mL/1-cm	1	2/pkg	48643-02

OPTIONAL REAGENTS

Chlorine Standard Solution, 2-mL Voluette® Ampule, 50–75 mg/L		20/pkg	14268-20
Potassium Iodide Solution, 30-g/L		100 mL MDB	343-32
Sodium Arsenite Solution, 5-g/L		100 mL MDB	1047-32
Sodium Hydroxide Standard Solution, 1.00 N		100 mL MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N		100 mL MDB	1270-32
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

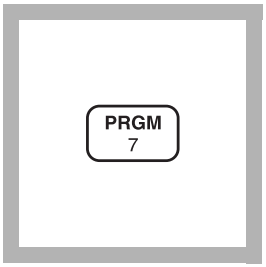
Ampule Breaker Kit		each	24846-00
Cylinder, graduated, 10-mL, mixing		each	20886-38
pH Meter, sens***ion***™1, portable, with electrode		each	51700-10
Pipet, TenSette®, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28

CHLORINE, FREE (0 to 2.00 mg/L)

DPD Method (Powder Pillows or AccuVac Ampuls)USEPA accepted for reporting wastewater and drinking water analyses*

Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*

Using Powder Pillows



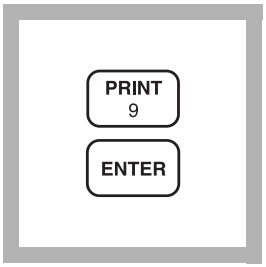
1. Enter the stored program number for free and total chlorine (Cl_2) powder pillows.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



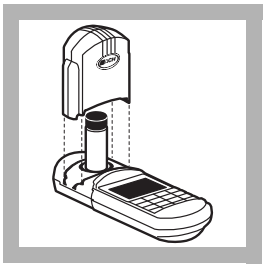
2. Press: **9 ENTER**

The display will show **mg/L, Cl2** and the **ZERO** icon.

3. Fill a sample cell with 10 mL of sample (the blank).

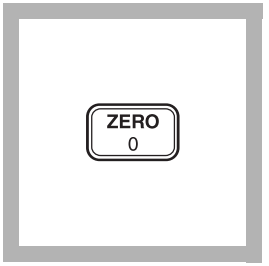
Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

Note: *The SwifTest Dispenser for Free Chlorine can be used in place of the powder pillows in step 7.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-Cl G for drinking water.

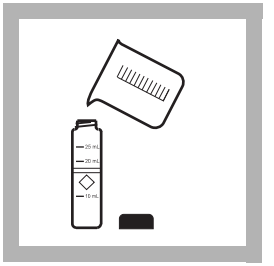


5. Press: **ZERO**

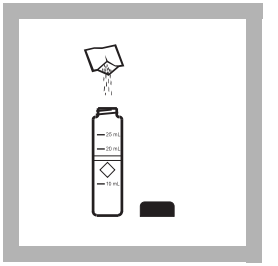
The cursor will move to the right, then the display will show:

0.00 mg/L Cl2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

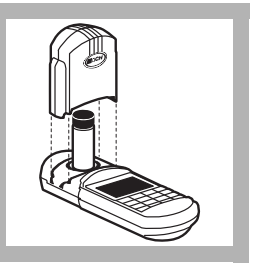


6. Fill another cell with 10 mL of sample.



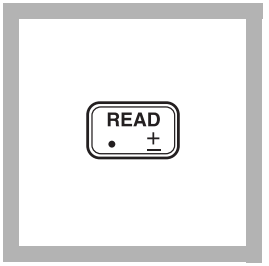
7. Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and swirl vigorously to dissolve the powder.

Note: *A pink color will develop if free chlorine is present.*



8. Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Perform Step 9 within one minute of reagent addition.*



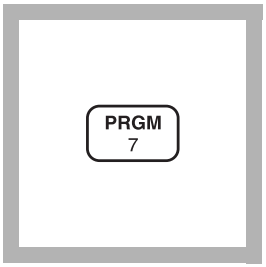
9. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Note: *If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the dilution factor; see Section 1. Or, use the High Range Free Chlorine test, program #8.*

Using AccuVac Ampuls



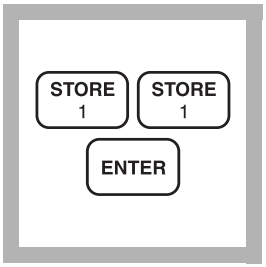
1. Enter the stored program number for free and total chlorine (Cl_2)-AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

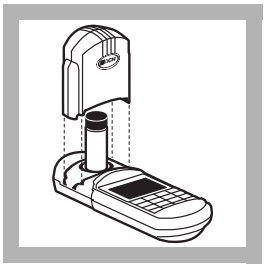


2. Press: **11 ENTER**

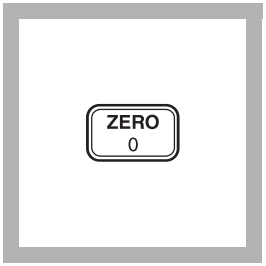
The display will show **mg/L, Cl2** and the **ZERO** icon.

3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

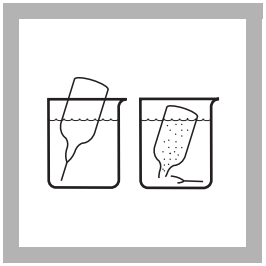


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

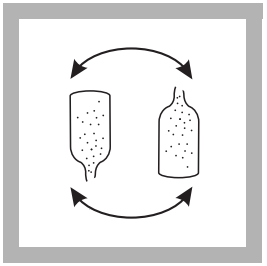
0.00 mg/L Cl2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



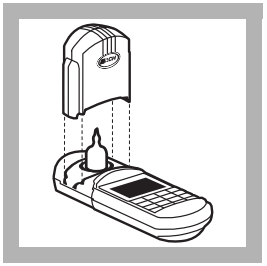
6. Fill a DPD Free Chlorine Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampule fills completely.*



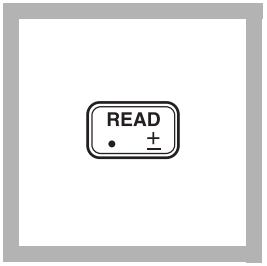
7. Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will form if chlorine is present.*



8. Immediately place the AccuVac Ampul into the cell holder. Tightly cover the ampule with the instrument cap.

Note: *Perform step 9 within one minute of reagent addition.*



9. Press: **READ**

The cursor will move to the right, then the result in mg/L chlorine will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Note: *If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the dilution factor; see Section 1.*

Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free chlorine is a strong oxidizing agent, and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of free chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

A common error in testing for chlorine is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the analysis immediately.

Accuracy Check

Standard Additions Method (using powder pillows)

a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.

b) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.

c) Re-zero the instrument using the original sample (the blank).

d) Place the spiked sample in the cell holder and press **READ**. Record the results.

e) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L Chlorine added} = \frac{0.1(\text{vol. standard added}) \times \text{Label value (mg/L } Cl_2)}{10.1(\text{sample + standard volume})}$$

f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step e).

g) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method (using AccuVac Ampuls)

a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.

b) Use a graduated cylinder to measure 25 mL of sample into each of two beakers.

c) Use a TenSette Pipet to add 0.2 mL of the standard to one of the beakers (this is the spiked sample). Swirl to mix.

d) Fill a DPD Free Chlorine AccuVac completely from each

beaker.

e) Analyze the spiked and unspiked sample as described in the procedure.

f) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L Chlorine added} = \frac{0.2(\text{vol. standard added}) \times \text{Label value (mg/L } Cl_2)}{25.2(\text{sample + standard volume})}$$

g) The spiked sample result should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step f).

h) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 1.00 mg/L chlorine and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of ±0.01 mg/L chlorine.

In a single laboratory using a standard solution of 1.00 mg/L chlorine and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.01 mg/L chlorine.

Estimated Detection Limit (EDL)

The estimated detection limit for programs 9 and 11 is 0.02 mg/L Cl_2. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium , Oxidized (Cr^{6+})	**1.** Adjust sample pH to 6-7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L increase in the reading.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See *Interferences, Section 1*.

Summary of Method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a magenta color which is proportional to the chlorine concentration.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
DPD Free Chlorine Powder Pillows, 10 mL	1 pillow	100/pkg	21055-69
Sample Cell, 10, 20, 25 mL, w/ cap	2	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

DPD Free Chlorine Reagent AccuVac Ampuls	1 ampul	25/pkg	25020-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Chlorine Standard Solution, PourRite ampule, 25-30 mg/L, 2 mL	20/pkg	26300-20
DPD Free Chlorine Reagent, SwifTest	250 tests	28023-00
Potassium Iodide Solution, 30 g/L	100 mL* MDB	343-32
Sodium Arsenite, 5 g/L	100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1.000 N	100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N	100 mL* MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Cylinder, graduated, 25 mL	each	508-40
pH Meter, *sens**ion***™***1***, portable, with electrode	each	51700-10
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96Pipet
Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
PourRite Ampule Breaker	each	24846-00

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Marked Dropper Bottle - contact Hach for larger sizes.

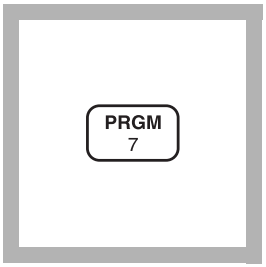
CHLORINE, TOTAL (0 to 2.00 mg/L)

DPD Method (Powder Pillows or AccuVac Ampuls)

USEPA accepted for reporting water and wastewater analyses*

Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*

Using Powder Pillows



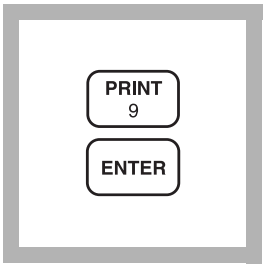
1. Enter the stored program number for total chlorine (Cl_2) powder pillows.

Press: **PRGM**

The display will show:

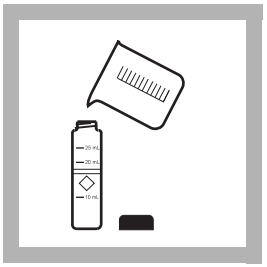
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



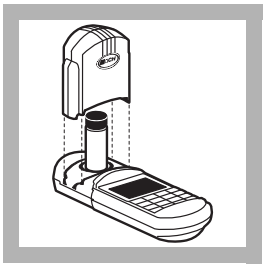
2. Press: **9 ENTER**

The display will show **mg/L, Cl2** and the **ZERO** icon**.**



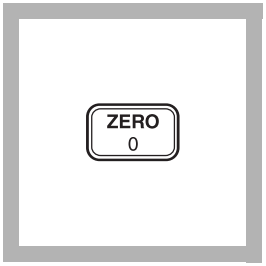
3. Fill a sample cell with 10 mL of sample (the blank).

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

* Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-Cl G for drinking water.

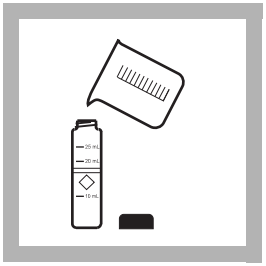


5. Press: **ZERO**

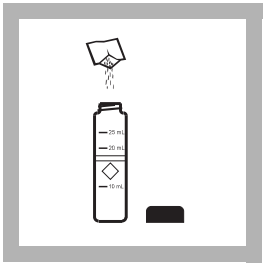
The cursor will move to the right, then the display will show:

0.00 mg/L Cl2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

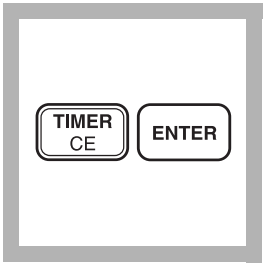


6. Fill a second cell to the 10-mL mark with sample.



7. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap and swirl the sample cell vigorously to dissolve the powder.

Note: *It is not necessary that all the powder dissolves.*

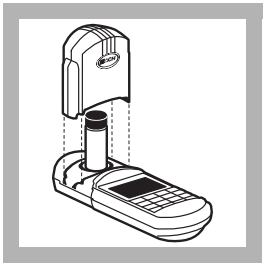


8. Press:

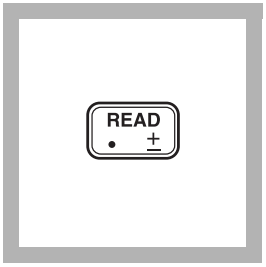
TIMER ENTER

A three-minute reaction period will begin. A pink color will develop if chlorine is present.

Note: *The SwifTest Dispenser for Total Chlorine can be used in place of the powder pillows in step 7.*



9. After the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



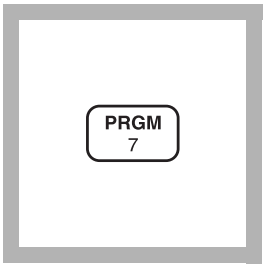
10. Press: **READ**

The cursor will move to the right, then the result in mg/L total chlorine will be displayed.

Note: *It the sample temporarily turns yellow after sample addition, or the display flashes "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the dilution factor; see Section 1. Or use the High Range Total Chlorine test, program #8.*

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Using AccuVac Ampuls



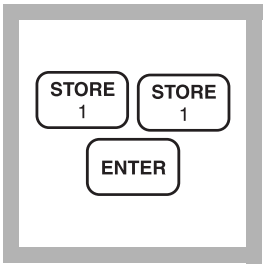
1. Enter the stored program number for total chlorine (Cl_2) AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

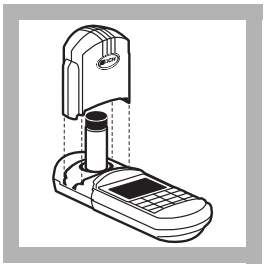


2. Press: **11 ENTER**

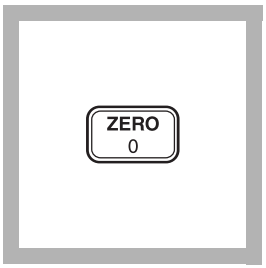
The display will show **mg/L, Cl2** and the **ZERO** icon**.**

3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

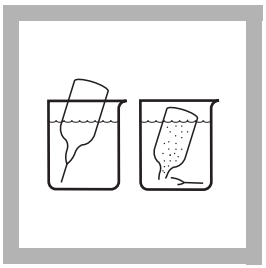


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

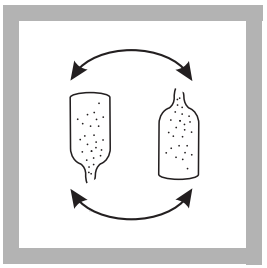
0.00 mg/L Cl2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



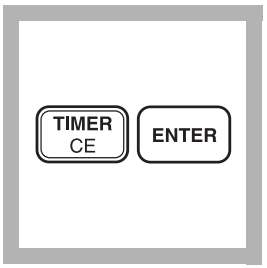
6. Fill a DPD Total Chlorine Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampule fills completely.*



7. Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.

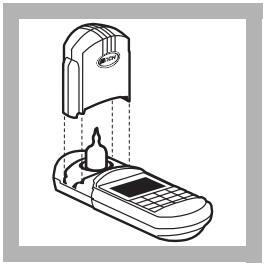
Note: *A pink color will form if chlorine is present.*



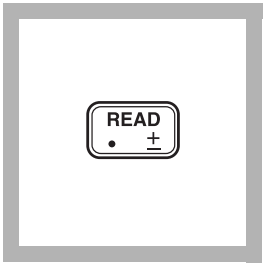
8. Press:

TIMER ENTER

A three-minute reaction period will begin.



9. When the timer beeps, place the AccuVac Ampul into the cell holder. Tightly cover the ampule with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L total chlorine will be displayed.

Note: *If the sample temporarily turns yellow after sample addition, or the display shows "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the appropriate dilution factor; see Section 1.*

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free chlorine is a strong oxidizing agent, and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

A common error in testing for chlorine is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the chlorine analysis immediately.

Accuracy Check

Standard Additions Method (using powder pillows)

a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.

b) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.

c) Re-zero the instrument using the original sample (the blank).

d) Place the spiked sample into the cell holder and press **READ**. Record the results.

e) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L chlorine added} = \frac{0.1\ (\text{vol. standard added}) \times \text{Label value (mg/L } Cl_2)}{10.1(\text{sample + standard volume})}$$

f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step e).

g) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method (using AccuVac Ampuls)

a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.

b) Use a graduated cylinder to measure 25 mL of sample into

each of two beakers.

c) Use a TenSette Pipet to add 0.2 mL of the standard to one of the beakers (this is the spiked sample). Swirl to mix.

d) Fill a DPD Total Chlorine AccuVac completely from each beaker.

e) Analyze the spiked and unspiked sample as described in
the procedure.

f) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L chlorine added} = \frac{0.2\ (\text{vol. standard added}) \times \text{Label value (mg/L Chlorine)}}{25.2\ (\text{sample + standard volume})}$$

g) The spiked sample result should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step f).

h) If this increase does not occur, see *Standard Additions* in *Section 1* for more information.

Method Performance

Precision

In a single laboratory, using a standard solution of 1.00 mg/L chlorine and two lots of reagents with the instrument, a single operator obtained standard deviations of ±0.01 mg/L chlorine.

In a single laboratory, using a standard solution of 1.00 mg/L chlorine and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.01 mg/L chlorine.

Estimated Detection Limit (EDL)

The estimated detection limit for programs 9 and 11 is 0.02 mg/L Cl_2. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions*).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium , Oxidized (Cr^{6+})	**1.** Adjust sample pH to 6-7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See *Interferences, Section 1*.

Summary of Method

Chlorine can be present in water as free available chlorine and as combined available chlorine. Both forms can exist in the same water and be determined together as the total available chlorine. Free chlorine is present as hypochlorous acid and/or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N, N-diethyl-p-phenylenediamine)

along with free chlorine present in the sample to form a red color which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run free chlorine and total chlorine tests. Subtract the results of the free chlorine test from the results of the total chlorine test to obtain combined chlorine.

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS & APPARATUS (USING POWDER PILLOWS)

Description	Qty/Test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows	1 pillow	100/pkg	21056-69
Sample Cell, 10-20-25 mL, w/caps	2	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (USING ACCUVAC AMPULS)

DPD Total Chlorine Reagent AccuVac Ampuls	1 ampul	25/pkg	25030-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Chlorine Standard Solution, PourRite ampule, 25-30 mg/L Cl_2	20/pkg	26300-20
DPD Total Chlorine Reagent, SwifTest	250 tests	28024-00
Potassium Iodide Solution, 30 g/L	100 mL* MDB	343-32
Sodium Arsenite, 5 g/L	100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1 N	100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1 N	100 mL* MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
PourRite Ampule Breaker	each	24846-00
Cylinder, graduated, 25 mL	each	508-40
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable	each	51700-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28

For Technical Assistance, Price and Ordering

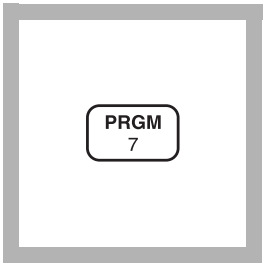
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Marked Dropper Bottle - contact Hach for larger sizes.

CHLORINE, FREE (0 to 5.00 mg/L)

DPD Test 'N Tube™ Method*

Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



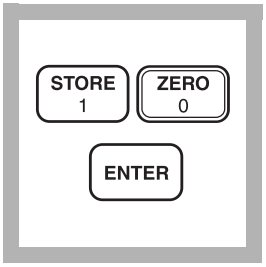
1. Enter the stored program number for Test 'N Tube free chlorine (Cl_2).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

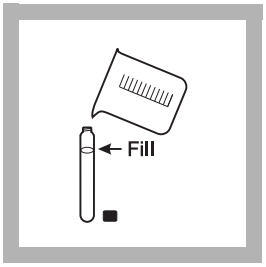


2. Press: **10 ENTER**

The display will show **mg/L, Cl2** and the **ZERO** icon**.**

3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down fully to insert it.

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

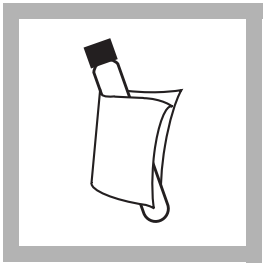


4. Fill an empty Test 'N Tube vial with sample (the blank).

Note: *Fill to the top of the Hach logo "oval" mark.*

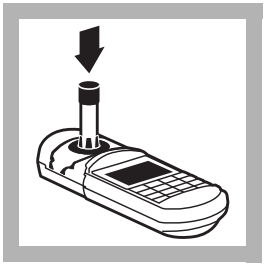
Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater*



5. Wipe the outside of the blank vial with a towel.

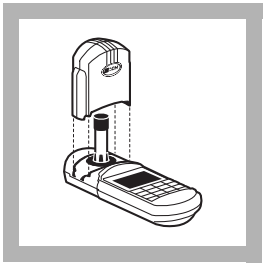
Note: *Wiping with a damp cloth followed by a dry one removes fingerprints and other marks.*



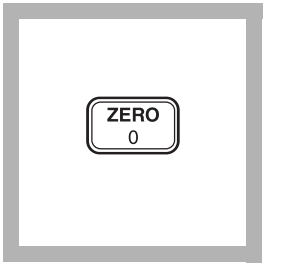
6. Place the blank in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



7. Cover the vial tightly with the instrument cap.

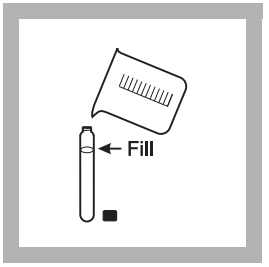


8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Cl2

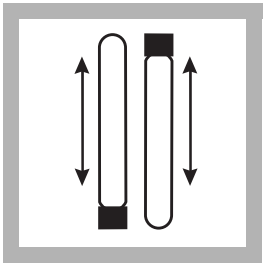
Note: *If Reagent Blank Correction is on, the display may show "limit". See Section 1.*



9. Remove the cap from a Free Chlorine DPD-TNT tube. Add 10 mL of sample.

Note: *Fill to the top of the Hach logo "oval" mark.*

Note: *A pink color will develop if chlorine is present.*



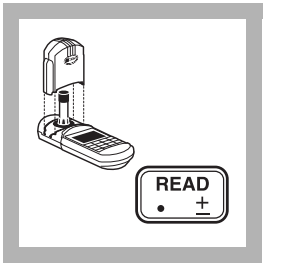
10. Cap and invert at least 10 times to dissolve the powder. This is the prepared sample.

Note: *Use slow, deliberate inversion for complete recovery. Ten inversions should take at least 30 seconds. One inversion equals turning the vial upside down, then returning it to an upright position.*

11. Within 30 seconds after mixing, wipe the prepared sample vial with a towel, then place it in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Cover the vial tightly with the instrument cap.

Press: **READ**

The cursor will move to the right, then the result in mg/L free chlorine will be displayed.

Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free chlorine is a strong oxidizing agent and is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature, and salinity influence decomposition of free chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pretreatment is necessary.

A common error in testing for chlorine is obtaining an unrepresentative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. Perform the analysis immediately.

Accuracy Check

Standard Additions Method

a) Snap the top off a HR Chlorine PourRite™ Ampule Standard Solution.

b) Use a TenSette® Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.

c) Analyze the spiked sample, beginning at Step 8 of the procedure.

d) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L chlorine added} = \frac{0.1(\text{vol. standard added}) \times \text{Label value}(\text{mg/L } Cl_2)}{10.1(\text{sample + standard volume})}$$

e) The spiked sample result (step c) should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step d).

f) If this increase does not occur, see *Standard Additions, Section 1* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 2.53 mg/L chlorine and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of ±0.14 mg/L chlorine.

Estimated Detection Limit (EDL)

The estimated detection limit for program 10 is 0.03 mg/L Cl_2. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1, Correcting for Volume Additions* in the *DR/800 Series Procedures Manual*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Section 1 Correcting for Volume Additions*).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium , oxidized (Cr^{6+})	**1.** Adjust sample pH to 6-7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.

<table>
<tr><th>Interfering Substance</th><th>Interference Level and Treatment</th></tr>
<tr><td>Monochloramine</td><td>For conventional free chlorine disinfection (beyond the breakpoint), typical monochloramine concentrations are very low. If monochloramine is present in the sample, its interference in the free chlorine test depends on the sample temperature, relative amount of monochloramine to free chlorine, and the time required to do the analysis. Typical interference level of monochloramine in the free chlorine test are listed below (as mg/L Cl_2).
<table>
<tr><th>NH_2Cl as Cl_2</th><th colspan="4">Sample Temp. °C (°F)</th></tr>
<tr><td></td><td>5 (40)</td><td>10 (50)</td><td>20 (68)</td><td>30 (83)</td></tr>
<tr><td>1.2 mg/L</td><td>+0.15</td><td>+0.19</td><td>+0.30</td><td>+0.29</td></tr>
<tr><td>2.5 mg/L</td><td>0.35</td><td>0.38</td><td>0.55</td><td>0.61</td></tr>
<tr><td>3.5 mg/L</td><td>0.38</td><td>0.56</td><td>0.69</td><td>0.73</td></tr>
</table>
</td></tr>
<tr><td>Ozone</td><td>Interferes at all levels</td></tr>
<tr><td>Peroxides</td><td>May interfere</td></tr>
<tr><td>Extreme sample pH and highly buffered samples</td><td>Adjust to pH 6-7. See Interferences, Section 1.</td></tr>
</table>

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). See *Section 3* for more information on proper disposal of these materials.

Summary of Method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD
(N,N-diethyl-p-phenylenediamine) indicator to form a magenta color which is proportional to the chlorine concentration.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Test 'N Tube DPD Free Chlorine Reagent	1 vial	50/pkg	21055-45
Test 'N Tube Vials	1 vial	6/pkg	22758-06

REQUIRED APPARATUS

Caps, white	1 cap	6/pkg	22411-06
COD/TNT Adapter	1	each	48464-00

OPTIONAL REAGENTS

Chlorine Standard Solution, PourRite ampule, 50-75 mg/L, 2 mL		20/pkg	14268-20
Potassium Iodide Solution, 30 g/L		100 mL* MDB	343-32
Sodium Arsenite, 5 g/L		100 mL* MDB	1047-32
Sodium Hydroxide Standard Solution, 1.000 N		100 mL* MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N		100 mL* MDB	1270-32

OPTIONAL APPARATUS

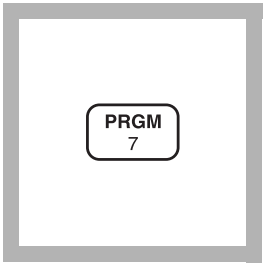
Beaker, 50 mL		each	500-41H
pH Meter, *sens**ion**™1*, portable, with electrode		each	51700-10
pH Paper, pH 1 to 11 pH		5 rolls/pkg	391-33
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite Ampule Breaker		each	24846-00
Test Tube Rack		each	18641-00

* Marked Dropper Bottle - contact Hach for larger sizes.

CHLORINE, TOTAL (0 to 5.00 mg/L)

DPD Test 'N Tube™ Method*

Note: *This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.*



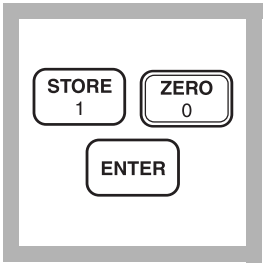
1. Enter the stored program number for Test 'N Tube total chlorine (Cl_2).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

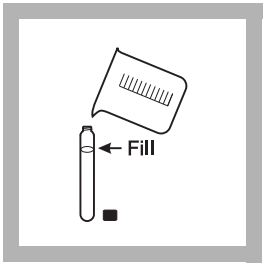


2. Press: **10 ENTER**

The display will show **mg/L, Cl2** and the **ZERO** icon**.**

3. Insert the COD/TNT Vial Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

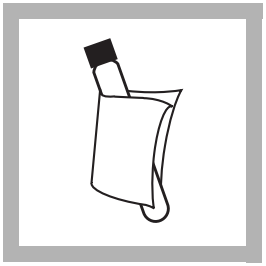


4. Fill an empty Test 'N Tube vial with sample (the blank).

Note: *Fill to the top of the Hach logo "oval" mark.*

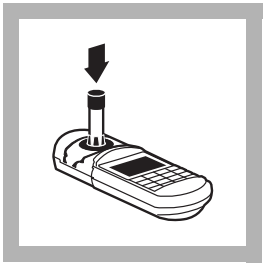
Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



5. Wipe the outside of the blank vial with a towel.

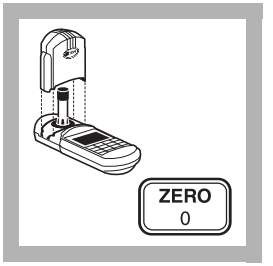
Note: *Wiping with a damp cloth followed by a dry one removes fingerprints and other marks.*



6. Place the blank in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



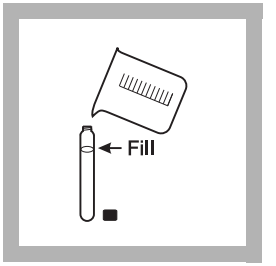
7. Cover the vial tightly with the instrument cap.

Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Cl2

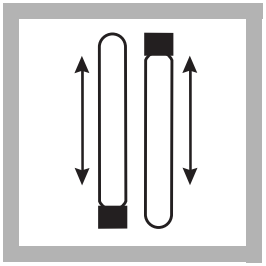
Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



8. Remove the cap from a Total Chlorine DPD-TNT tube. Add 10 mL of sample.

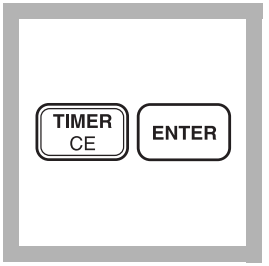
Note: *Fill to the top of the Hach logo "oval" mark.*

Note: *A pink color will develop if chlorine is present.*



9. Cap and invert at least 10 times to dissolve the powder. This is the prepared sample.

Note: *Use slow, deliberate inversion for complete recovery. Ten inversions should take at least 30 seconds. One inversion equals turning the vial upside down, then returning it to an upright position.*

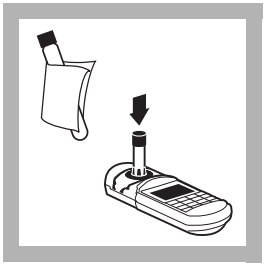


10. Press:

TIMER ENTER

A three-minute reaction period will begin.

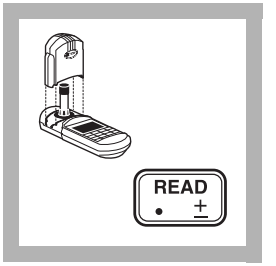
Note: *A pink color will develop if chlorine is present.*



11. When the timer beeps, wipe the prepared sample vial with a towel, then place it in the vial adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Cover the vial tightly with the instrument cap.

Press: **READ**

The cursor will move to the right, then the result in mg/L total chlorine will be displayed.

Sampling and Storage

Analyze samples for chlorine **immediately** after collection. Free and combined chlorine are strong oxidizing agents and are unstable in natural waters. They react rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of chlorine in water.

Avoid plastic containers since these may have a large chlorine demand. **Pretreat glass** sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to l liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

A common error in testing for chlorine is obtaining an unrepresentative sample. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample containers so there is no headspace (air) above the sample. Perform the analysis immediately.

Accuracy Check

Standard Additions Method

a) Snap the top off a High Range Chlorine PourRite™ Ampule Standard Solution.

b) Use a TenSette® Pipet to add 0.1 mL of the standard to 10 mL of sample (this is the spiked sample). Swirl to mix.

c) Analyze the spiked sample, beginning at Step 8 of the procedure.

d) Calculate the concentration of mg/L chlorine added to the sample:

$$\text{mg/L chlorine added} = \frac{0.1\ (\text{vol. standard added}) \times \text{Label value (mg/L } Cl_2)}{10.1(\text{sample + standard volume})}$$

e) The spiked sample result (step c) should reflect the analyzed sample result + the calculated mg/L Cl_2 added (step d).

f) If this increase does not occur, see *Standard Additions, Section 1* for more information.

Method Performance

Precision

In a single laboratory, using a standard solution of 2.53 mg/L chlorine and two representative lots of reagents with the instrument, a single operator obtained standard deviations of ±0.14 mg/L chlorine.

Estimated Detection Limit (EDL)

The estimated detection limit for programs 10 is 0.03 mg/L Cl_2. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Correcting for Volume Additions* in *Section 1*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition (See *Correcting for Volume Additions* in *Section 1*).
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Iodine	Interferes at all levels
Manganese, oxidized (Mn^{4+}, Mn^{7+}) or Chromium , oxidized (Cr^{6+})	**1.** Adjust sample pH to 6-7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait one minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7. See *Interferences* in *Section 1*.

Summary of Method

Chlorine can be present in water as free available chlorine and as combined available chlorine. Both forms can exist in the same water and be determined together as the total available chlorine. Free chlorine is present as hypochlorous acid and/or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N, N-diethyl-p-phenylenediamine) along with free chlorine present in the sample to form a red color which is proportional to the total chlorine concentration. To determine the concentration of combined chlorine, run free chlorine and total chlorine tests. Subtract the results of the free chlorine test from the results of the total chlorine test to obtain combined chlorine.

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004).

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Test 'N Tube DPD Total Chlorine Reagent	1 vial	25/pkg	21056-25
Test 'N Tube Vials	1 vial	6/pkg	22758-06

REQUIRED APPARATUS

COD/TNT Adapter, DR/800	1	each	48464-00

OPTIONAL REAGENTS

Chlorine Standard Solution, 2-mL PourRite ampule, 50-75 mg/L	20/pkg		14268-20
Potassium Iodide Solution, 30 g/L	100 mL*	MDB	343-32
Sodium Arsenite Solution, 5 g/L	100 mL*	MDB	1047-32
Sodium Hydroxide Standard Solution, 1.00 N	100 mL*	MDB	1045-32
Sulfuric Acid Standard Solution, 1.000 N	100 mL*	MDB	1270-32

OPTIONAL APPARATUS

Beaker, 50 mL	each		500-41H
PourRite Ampule Breaker	each		24846-00
pH Indicator Paper, pH 1 to 11	5 rolls/pkg		391-33
pH Meter, *sens**ion***™***1**, portable, with electrode	each		51700-10
Pipet, TenSette, 0.1 to 1.0 mL	each		19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg		21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg		21856-28
Test Tube Rack	each		18641-00

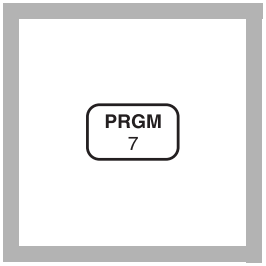
* Marked Dropper Bottle - contact Hach for larger sizes.

CHROMIUM, HEXAVALENT (0 to 0.60 mg/L Cr^{6+}) For water and wastewater

1,5-Diphenylcarbohydrazide Method* (Powder Pillows or AccuVac Ampuls)

USEPA accepted for wastewater analyses**

Using Powder Pillows

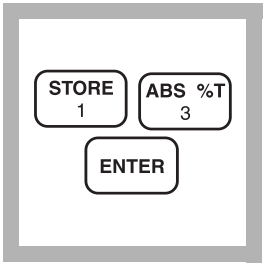


1. Enter the stored program number for hexavalent chromium (Cr^{6+})- powder pillows.

Press: **PRGM**

The display will show:

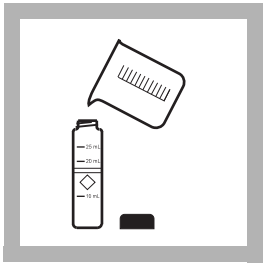
PRGM ?



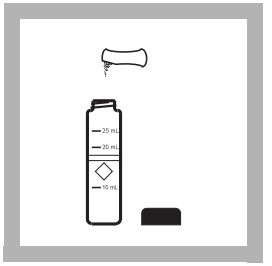
2. Press: **13 ENTER**

The display will show **mg/L, Cr6** and the **ZERO** icon**.**

Note: *For alternate forms (CrO_4, Cr_2O_7), press the* **CONC** *key.*

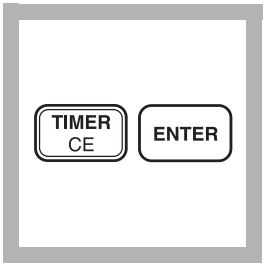


3. Fill a sample cell with 10 mL of sample.



4. Add the contents of one ChromaVer 3 Reagent Powder Pillow to the cell (the prepared sample). Cap the cell and invert several times to mix.

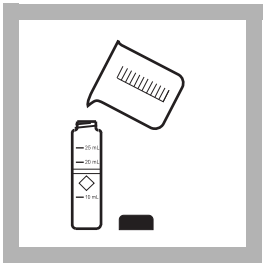
Note: *A purple color will form if Cr^{6+} is present.*



5. Press:

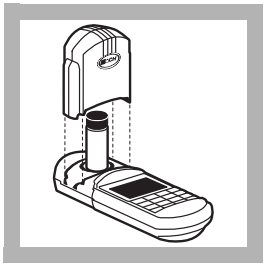
TIMER ENTER

A five-minute reaction period will begin.

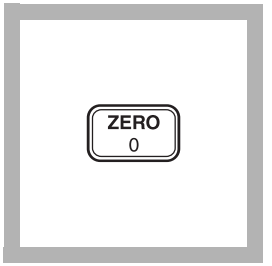


6. Fill another sample cell with 10 mL of sample (the blank).

Note: *For turbid samples, add the contents of one Acid Reagent Powder Pillow. This ensures turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent is also dissolved in the blank.*



7. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **ZERO**

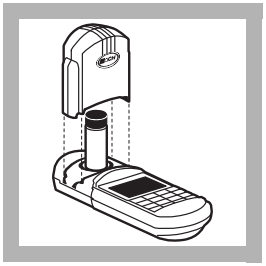
The cursor will move to the right, then the display will show:

0.00 mg/L Cr6

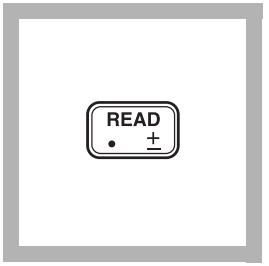
---

* Adapted from *Standard Methods for the Examination of Water and Wastewater*

** Procedure is equivalent to USGS method I-1230-85 for wastewater.



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

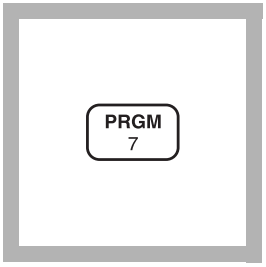


10. Press: **READ**

The cursor will move to the right, then the result in mg/L hexavalent chromium will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Using Accuvac Ampuls

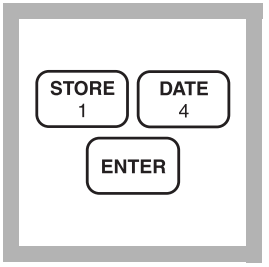


1. Enter the stored program number for hexavalent chromium (Cr^{6+})- AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?



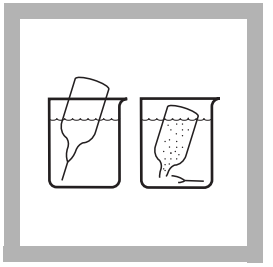
2. Press: **14 ENTER**

The display will show **mg/L, Cr6** and the **ZERO** icon**.**

Note: *For alternate forms (CrO_4, Cr_2O_7), press the* **CONC** *key.*

3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

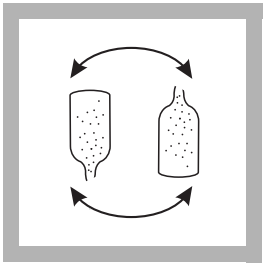
Note: *For turbid samples, add the contents of one Acid Reagent Powder Pillow to 10 mL of the blank. This ensures turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent is also dissolved in the blank.*



4. Fill a ChromaVer 3 Reagent AccuVac Ampul (the prepared sample) with sample.

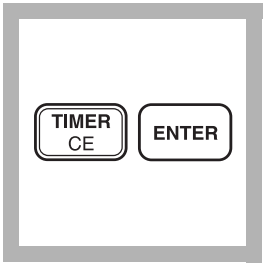
Note: *Keep the tip immersed while the ampul fills completely.*

Note: *ChromaVer 3 should be white to tan in color. Replace if it is brown or green.*



5. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

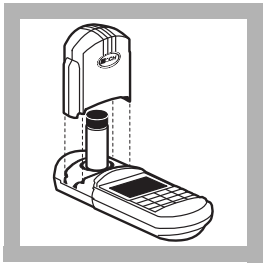
Note: *A purple color will form if hexavalent chromium is present.*



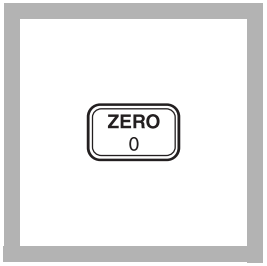
6. Press:

TIMER ENTER

A five-minute reaction period will begin.



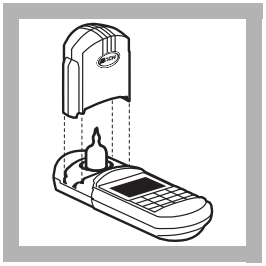
7. When the timer beeps place the blank into the cell holder.



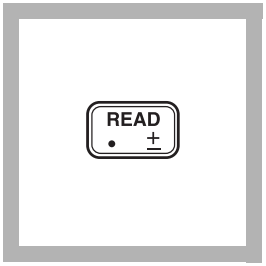
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Cr6



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L hexavalent chromium will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in a cleaned glass or plastic container. Store at 4 °C (39 °F) up to 24 hours. Samples must be analyzed within 24 hours.

Accuracy Check

Standard Additions Method (powder pillows)

a) Snap the neck off a Hexavalent Chromium PourRite Standard Ampule, 5 mg/L Cr^{6+}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 10-mL samples, respectively. Swirl to mix.

c) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method (AccuVac Ampuls)

a) Snap the neck off a Hexavalent Chromium Voluette Standard Ampule, 12.5 mg/L Cr^{6+}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 25-mL samples in beakers. Swirl gently to mix.

c) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 0.50-mg/L Cr^{6+} solution by pipetting 10.00 mL of Hexavalent Chromium Standard Solution, 50.0 mg/L Cr^{6+}, into a 1000-mL volumetric flask and diluting to the mark with deionized water. Invert repeatedly to mix. Prepare this solution daily. Perform the chromium procedure as described above, using this solution in place of the sample.

Method Performance

Precision

In a single laboratory using a standard solution of 0.6 mg/L Cr^{6+} and two representative lots of powder pillow reagent with the instrument, a single operator obtained a standard deviation of ±0.008 mg/L Cr^{6+}.

In a single laboratory using a standard solution of 0.6 mg/L Cr^{6+} and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.005 mg/L Cr^{6+}.

Estimated Detection Limit (EDL)

The EDL for program 13 (powder pillows) and program 14 (AccuVac Ampuls) is 0.01 mg/L Cr^{6+}. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

The following substances do not interfere in the test, up to the following concentration:

Substance	Concentration
Mercurous & Mercuric Ions	Interferes slightly
Iron	1 mg/L
Vanadium	1 mg/L. At higher levels vanadium interference can be overcome by waiting ten minutes before reading.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interference* in *Section 1*.

Summary of Method

Hexavalent chromium is determined by the 1,5-diphenylcarbohydrazide method using a single dry powder formulation called ChromaVer 3 Chromium Reagent. This reagent contains an acidic buffer combined with 1,5-diphenylcarbohydrazide, which reacts to give a purple color which is proportional to the amount of hexavalent chromium present.

REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
ChromaVer 3 Chromium Reagent Powder Pillows..	1 pillow	100/pkg	12710-99
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

REQUIRED REAGENTS AND APPARATUS (Using AccuVac Ampuls)

ChromaVer 3 AccuVac Ampuls	1 ampul	25/pkg	25050-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Description	Unit	Cat. No
Acid Reagent Powder Pillows	100/pkg	2126-99
Chromium, Hexavalent, Standard Solution, 50 mg/L Cr^{6+}	100 mL	810-42
Chromium, Hexavalent, Standard Solution, Voluette Ampule, 12.5 mg/L Cr^{6+}, 10 mL	16/pkg	14256-10
Chromium, Hexavalent, Standard Solution, PourRite Ampule, 5 mg/L Cr^{6+}, 2 mL	20/pkg	26056-20
Water, deionized	4 L	272-56

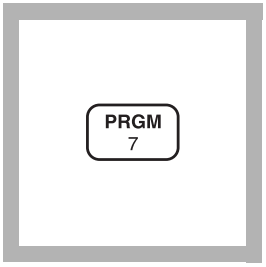
OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, EC10, portable	each	50050-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 5.00 mL, Class A	each	14515-37
Pipet Filler, safety bulb	each	14651-00
PourRite Ampule Breaker, 2 mL	each	24846-00

In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

CHROMIUM, TOTAL (0 to 0.60 mg/L)

Alkaline Hypobromite Oxidation Method* **



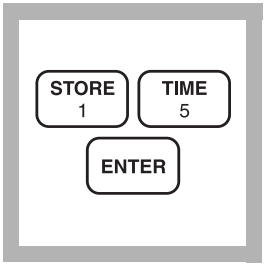
1. Enter the stored program number for total chromium (Cr).

Press: **PRGM**

The display will show:

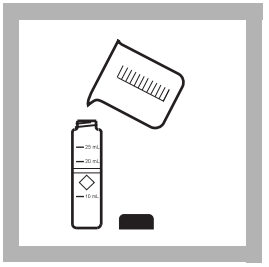
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



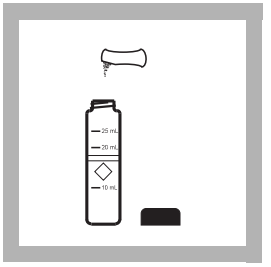
2. Press: **15 ENTER**

The display will show **mg/L, Cr** and the **ZERO** icon**.**

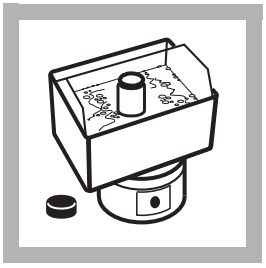


3. Fill a clean sample cell with 25 mL of sample.

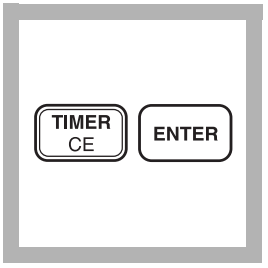
Note: *Adjust the pH of stored samples before analysis.*



4. Add the contents of one Chromium 1 Reagent Powder Pillow (the prepared sample). Cap the cell and invert repeatedly to mix. Remove the cap.



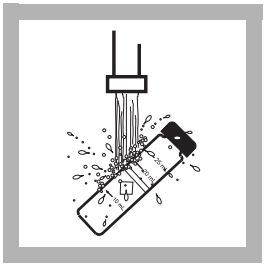
5. Place the prepared sample into a boiling water bath.



6. Press:

TIMER ENTER

A five-minute reaction period will begin.



7. After the beeper beeps, remove the prepared sample. Cap the cell. Use running tap water to cool the cell to 25 °C.

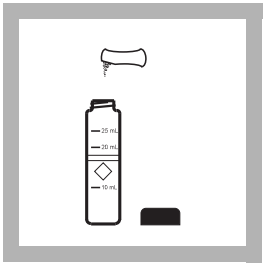
Note: *Use finger cots to handle the hot sample cell.*

8. Add the contents of one Chromium 2 Reagent Powder Pillow. Cap the cell and invert repeatedly to mix. Remove the cap.

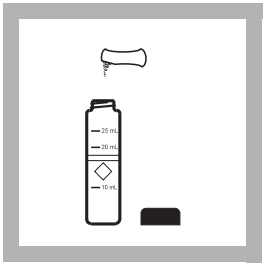
---

* Adapted from *Standard Methods for the Examination of Water and Wastewater*

** Procedure is equivalent to Standard Method 3500-Cr D for wastewater.



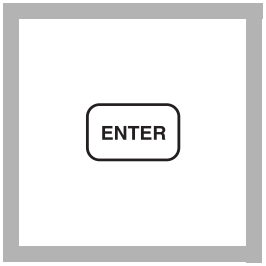
9. Add the contents of one Acid Reagent Powder Pillow. Cap the cell and invert repeatedly to mix. Remove the cap.



10. Add the contents of one ChromaVer 3 Chromium Reagent Powder Pillow. Cap the cell and invert repeatedly to mix.

Note: *A purple color will form if chromium is present.*

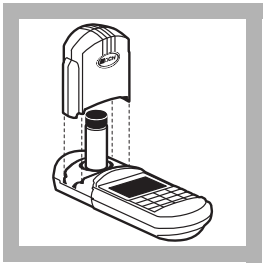
Note: *ChromaVer 3 is white to tan in color. Replace brown or green powder. Undissolved powder does not affect accuracy.*



11. The display will show: **05:00 TIMER 2**

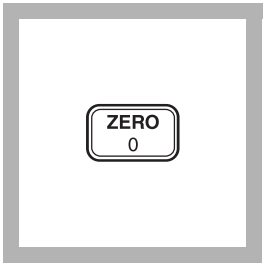
Press: **ENTER**

A five-minute reaction period will begin.



12. After the timer beeps, fill another sample cell with 25 mL of sample (the blank). Place it into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *For turbid samples, treat the blank as a sample, adding all reagents except the ChromaVer 3.*

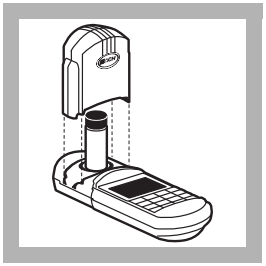


13. Press: **ZERO**

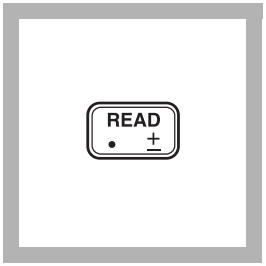
The cursor will move to the right, then the display will show:

0.00 mg/L Cr

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



14. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



15. Press: **READ**

The cursor will move to the right, then the result in mg/L total chromium (Cr) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-washed glass or plastic containers. To preserve samples, adjust the pH to 2 or lower with nitric acid (about 2 mL per liter). Store preserved samples at room temperature up to six months. Adjust the pH to about 4 with 5.0 N Sodium Hydroxide before analysis. Correct the test results for volume additions (see *Section 1*).

Accuracy Check

Standard Additions Method

a) Fill three sample cells with 25 mL of sample.

b) Snap the top off a Trivalent Chromium Standard Ampule, 12.5 mg/L as Cr^{3+}.

c) Use the TenSette pipet to add 0.1, 0.2, and 0.3 mL of standard to the three sample cells. Cap and invert repeatedly to mix .

d) Analyze each sample as described above. The chromium concentration should increase 0.05 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur see *Standard Additions* (*Section 1*).

Standard Solution Method

Prepare a 0.5 mg/L trivalent chromium standard by diluting 1.00 mL of Trivalent Chromium Standard Solution, 50 mg/L as Cr^{3+}, to 100 mL with deionized water. Mix thoroughly. Prepare this solution daily. Perform the chromium procedure as described above. The mg/L Cr reading should be 0.5 mg/L.

Method Performance

Precision

In a single laboratory using a standard solution of 0.4 mg/L trivalent chromium and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.004 mg/L chromium.

Estimated Detection Limit

The estimated detection limit for program 15 is 0.01 mg/L Cr. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Suggested Treatment
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interferences* in *Section 1*.
Large amounts of organic material	May inhibit complete oxidation of trivalent chromium. If high levels of organic material are present, see *Digestion* in *Section 2* for instruction on sample digestion. Perform the analysis as described on the digested sample.

Summary of Method

Trivalent chromium in the sample is oxidized to the hexavalent form by hypobromite ion under alkaline conditions. The sample is acidified. The total chromium content is determined by the 1,5-diphenylcarbohydrazide method. Determine trivalent chromium by subtracting the results of a separate hexavalent chromium test from the results of the total chromium test.

REQUIRED REAGENTS

	Cat. No.
Total Chromium Reagent Set (100 Tests)	22425-00
Includes: (1) 2126-99, (1) 12066-99, (1) 2043-99, (1) 2044-99	

Description	Quantity Required Per Test	Unit	Cat. No.
Acid Reagent Powder Pillows	1 pillow	100/pkg	2126-99
ChromaVer 3 Chromium Reagent Powder Pillows	1 pillow	100/pkg	12066-99
Chromium 1 Reagent Powder Pillows	1 pillow	100/pkg	2043-99
Chromium 2 Reagent Powder Pillows	1 pillow	100/pkg	2044-99

REQUIRED APPARATUS

Hot plate, 4" diameter, 120 V	1	each	12067-01
OR			
Hot plate, 4" diameter, 240 V	1	each	12067-02
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06
Water bath and rack	1	each	1955-55

OPTIONAL REAGENTS

Chromium, trivalent, Standard Solution, 50 mg/L Cr^{3+}		100 mL	14151-42
Chromium, trivalent, Standard Solution, PourRite ampule, 12.5 mg/L Cr^{3+}, 10 mL		16/pkg	14257-10
Nitric Acid, ACS		500 mL	152-49
Nitric Acid Solution 1:1		500 mL	2540-49
Sodium Hydroxide Standard Solution 5.0 N		50 mL* DB	2450-26
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Cylinder, graduated, polypropylene, 25 mL		each	1081-40
Finger Cots		2/pkg	14647-02
pH Paper, 1 to 11 pH units		5 rolls/pkg	391-33
pH Meter, sens*ion 1*, with electrode		each	51700-10
Pipet, serological, 2 mL		each	532-36
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet, volumetric, Class A, 1.00 mL		each	14515-35
Pipet Filler, safety bulb		each	14651-00
Ampule Breaker, 10-mL		each	21968-00

For Technical Assistance, Price and Ordering

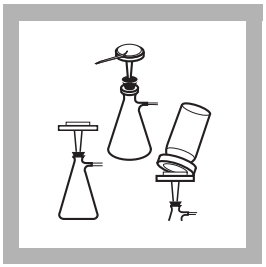
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes.

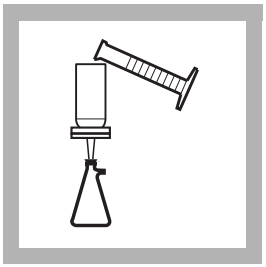
COLOR, TRUE AND APPARENT (0 to 500 units)

APHA Platinum-Cobalt Standard Method* For water, wastewater and seawater

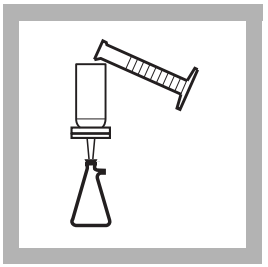


1. Assemble the filtering apparatus (membrane filter, filter holder, filter flask, and aspirator).

Note: *To test for apparent color, do not filter; begin at Step 4 and skip Step 7.*



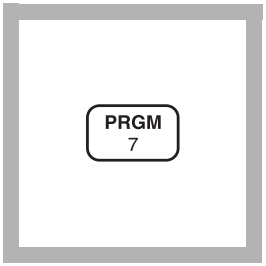
2. Rinse the filter by pouring about 50 mL of deionized water through the filter. Discard the rinse water.



3. Pour another 50 mL of deionized water through the filter. Keep this for Step 4.

4. Fill a sample cell (the blank) with 25 mL of filtered deionized water. Discard the excess.

Note: *For apparent color use unfiltered deionized water.*

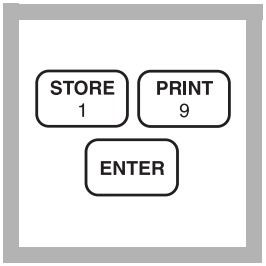


5. Enter the stored program number for APHA color.

Press: **PRGM**

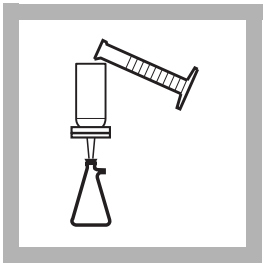
The display will show:

PRGM ?



6. Press: **19 ENTER**

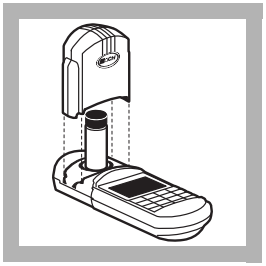
The display will show **PtCo** and the **ZERO** icon**.**



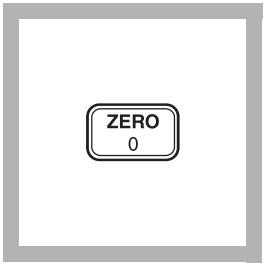
7. Pour about 50 mL of sample through the filter.

8. Fill a second sample cell (the prepared sample) with 25 mL of the filtered sample.

* Adapted from *Standard Methods for the Examination of Water and Wastewater*



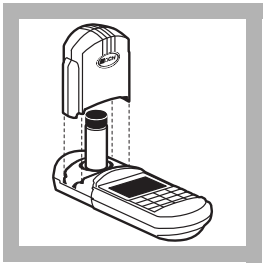
9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



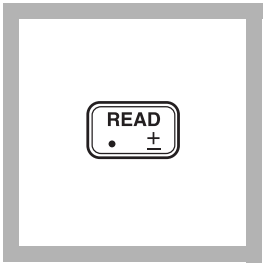
10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L Pt Co



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in Platinum-Cobalt color units (Pt-Co) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze the sample as soon as possible after collection for best results. If prompt analysis is impossible, fill bottles completely and cap tightly. Avoid excessive agitation or prolonged contact with air. Samples can be stored for 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before running the test.

Accuracy Check

Standard Solution Method

A 500 Platinum-Cobalt Units Color Standard solution is available for checking test accuracy. A 250 Platinum-Cobalt Units Standard can be made by pipetting 50.0 mL of the 500 Platinum-Cobalt Units Standard into a 100-mL volumetric flask and diluting to volume with deionized water.

Method Performance

Precision

In a single laboratory, using a standard solution of 250 Pt-Co color units and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±10 Pt-Co color units. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 19 is 25 Pt-Co color units. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

Color may be expressed as “apparent” or “true” color. The apparent color includes color from dissolved materials plus that from suspended matter. By filtering or centrifuging out the suspended materials, the true color can be determined. The procedure describes true color analysis. If apparent color is desired, it can be determined by measuring an unfiltered water sample. The stored program is used for both forms of color.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Units	Cat. No.
Water, deionized	50 mL	4 L	272-56

REQUIRED APPARATUS

Aspirator, vacuum	1	each	2131-00
Filter Holder, 47 mm, 300 mL graduated	1	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	1	100/pkg	13530-00
Flask, filtering, 500 mL	1	each	546-49
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
Stopper, No. 7, one hole	1	6/pkg	2119-07

OPTIONAL REAGENTS

Color Standard Solution, 500 platinum-cobalt units		1 L	1414-53

OPTIONAL APPARATUS

Cylinder, graduated, 50-mL, glass		each	508-41
Flask, volumetric, Class A, 100 mL		each	14574-42
Pipet, volumetric, Class A, 50 mL		each	14515-41
Thermometer, –20 to 110 °C, non-mercury		each	26357-02

For Technical Assistance, Price and Ordering

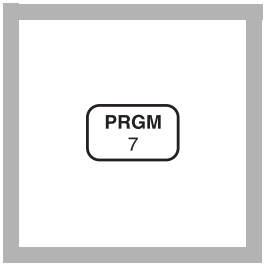
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

COPPER (0 to 5.00 mg/L)

For water, wastewater and seawater*

Bicinchoninate Method** (Powder Pillows or AccuVac Ampuls); USEPA approved for reporting wastewater analysis (digestion needed; See Section 2)*** Using Powder Pillows



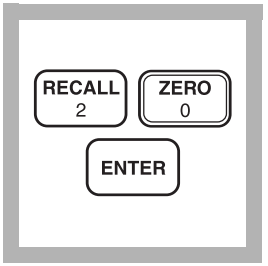
1. Enter the stored program number for bicinchoninate copper (Cu)- powder pillows.

Press: **PRGM**

The display will show:

PRGM ?

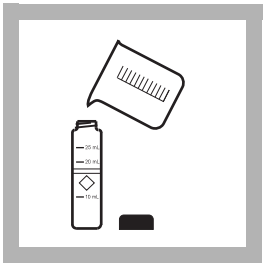
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **20 ENTER**

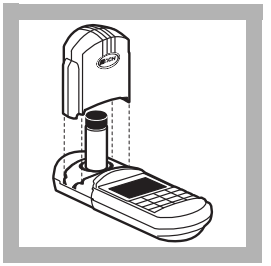
The display will show **mg/L, Cu** and the **ZERO** icon**.**

Note: *Determination of total copper needs a prior digestion (see Digestion in Section 2).*



3. Fill a sample cell with 10 mL of sample (the blank).

Note: *Adjust the pH of acid-preserved samples to 4-6 with 8 N KOH before analysis. Do not exceed pH 6 or copper may precipitate.*

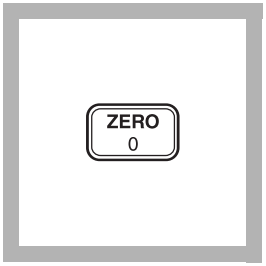


4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

* Pretreatment required; see *Interferences (Using Powder Pillows)*

** Adapted from Nakano, S., *Yakugaku Zasshi*, 82 486-491 (1962) [*Chemical Abstracts*, 58 3390e (1963)]

*** Powder Pillows only: *Federal Register*, 45 (105) 36166 (May 29, 1980)

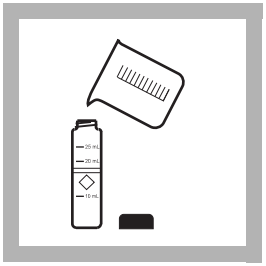


5. Press: **ZERO**

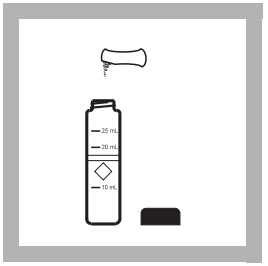
The cursor will move to the right, then the display will show:

0.00 mg/L Cu

Note: *If Reagent Blank Correction is on, the display may flash "limit".*

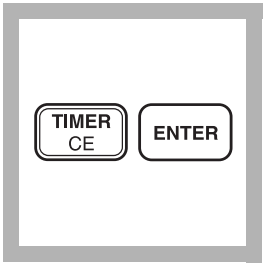


6. Fill another sample cell with 10 mL of the sample.



7. Add the contents of one CuVer 1 Copper Reagent Powder Pillow to the sample cell (the prepared sample). Swirl the cell to mix.

Note: *If copper is present, A purple color will develop.*

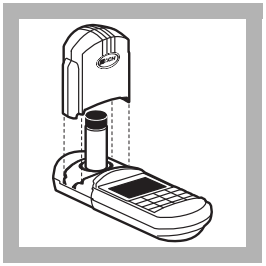


8. Press:

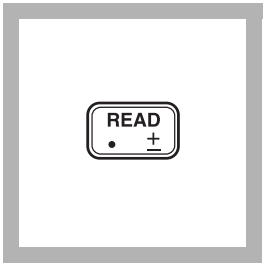
TIMER ENTER

A two-minute reaction period will begin.

Note: *Accuracy is not affected by undissolved powder.*



9. Within 30 minutes after the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



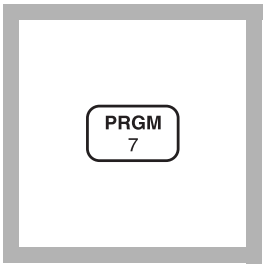
10. Press: **READ**

The cursor will move to the right, then the result in mg/L copper will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Using AccuVac Ampuls

Method 8026



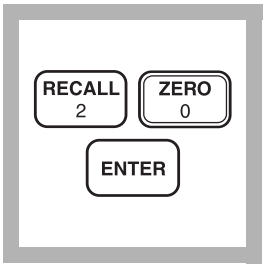
1. Enter the stored program number for bicinchoninate copper (Cu)- AccuVac ampuls.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



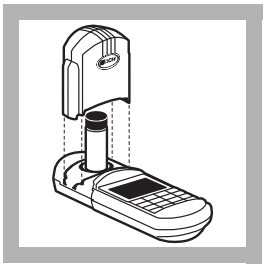
2. Press: **20 ENTER**

The display will show **mg/L, Cu** and the **ZERO** icon**.**

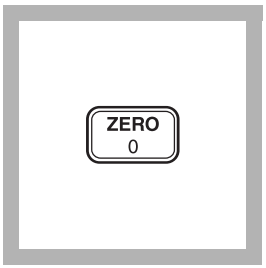
Note: *Determination of total copper needs a prior digestion (see Digestion in Section 2).*

Note: *Adjust the pH of stored samples before analysis.*

3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

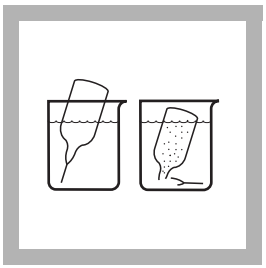


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

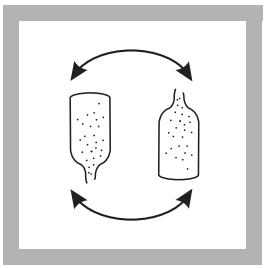
0.00 mg/L Cu

Note: *If Reagent Blank Correction is on, the display may flash "limit".*



6. Fill a CuVer 2 Copper Reagent AccuVac Ampul with sample.

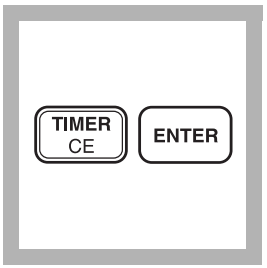
Note: *Keep the tip immersed while the ampul fills completely.*



7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A purple color will form if copper is present.*

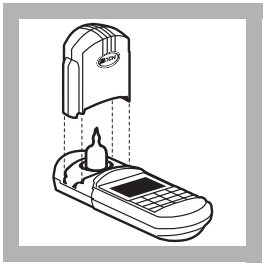
Note: *Accuracy is not affected by undissolved powder*



8. Press:

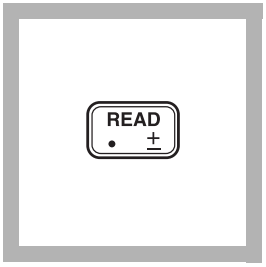
TIMER ENTER

A two-minute reaction period will begin.



9. After the timer beeps, place the AccuVac ampul in the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Step 10 must be completed within 30 minutes after the timer beeps.*



10. Press: **READ**

The cursor will move to the right, then the result in mg/L copper (Cu) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see in Section 1).*

Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Store preserved samples up to six months at room temperature. Before analysis, adjust the pH to 4 to 6 with 8 N potassium hydroxide. Do not exceed pH 6, as copper may precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information. If only dissolved copper is to be determined, filter the sample before acid addition using the labware listed under *Optional Apparatus*.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Copper Voluette Ampule Standard, 75 mg/L as Cu.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to the mixing cylinders. Stopper and mix thoroughly.

d) For analysis with AccuVac Ampuls, transfer the solutions to dry, clean 50-mL beakers to fill the ampules. For analysis with powder pillows, transfer only 10 mL of the solution to 10-mL sample cells.

e) Analyze each sample as described in the procedure. The copper concentration should increase about 0.3 mg/L for each 0.1 mL of standard added.

f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 1.00 mg/L copper standard by pipetting 1.00 mL of Copper Standard Solution, 100 mg/L as Cu, into 100-mL volumetric flask. Dilute to volume with deionized water and mix well. Prepare this solution daily. Using this solution as the sample, perform the copper procedure as described above.

Method Performance

Precision

In a single laboratory, using a standard solution of 2.25 mg/L Cu

and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.02 mg/L Cu.

In a single laboratory, using a standard solution of 2.25 mg/L Cu and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.02 mg/L Cu.

Estimated Detection Limit (EDL)
The EDL for program 20 (Powder Pillows and AccuVac Ampuls) is 0.02 mg/L Cu. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatments for Powder Pillows

Interfering Substance	Interference Level and Treatment
Acidity	If the sample is extremely acidic (pH 2 or less) a precipitate may form. Add 8 N Potassium Hydroxide Standard Solution drop-wise while swirling to dissolve the turbidity. Continue with Step 3.
Aluminum, Al^{3+}	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in Step 4. Results obtained will include total dissolved copper (free and complexed).
Cyanide, CN^-	Prevents full color development. Add 0.2 mL of formaldehyde to the 10-mL sample. Wait 4 minutes before taking the reading. Multiply the test results by 1.02 to correct for sample dilution by the formaldehyde.
Hardness	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in Step 4. Results obtained will include total dissolved copper (free and complexed).
Iron, Fe^{3+}	Follow the powder pillow procedure above, but substitute a CuVer 2 Copper Reagent Powder Pillow for the CuVer 1 Pillow used in Step 4. Results obtained will include total dissolved copper (free and complexed).
Silver, Ag^+	If a turbidity remains and the precipitate turns black, silver interference is likely. Add 10 drops of saturated Potassium Chloride Solution to 75 mL of sample, followed by filtering through a fine or highly retentive filter. Use the filtered sample in the procedure.

To differentiate free copper from that complexed to EDTA or other complexing agents, use a Free Copper Reagent Powder

Pillow in place of the CuVer 1 pillow in Step 4. Results in Step 10 will be free copper only. Add a Hydrosulfite Reagent Powder Pillow to the same sample and re-read the result. This result will include the total dissolved copper (free and complexed).

Interfering Substances and Suggested Treatments for AccuVac Ampuls

Interfering Substance	Interference Level and Treatment
Acidity	If the sample is extremely acidic (pH 2 or less) a precipitate may form. Add 8 N Potassium Hydroxide Standard Solution drop-wise until sample pH is above 4. Continue with Step 3.
Aluminum, Al^{3+}	Reagents accommodate high levels.
Cyanide, CN^-	Prevents full color development. Add 1.0 mL of formaldehyde to a 50-mL sample. Wait 4 minutes before taking the reading. Multiply the test results by 1.02 to correct for sample dilution by the formaldehyde.
Hardness	Reagents accommodate high levels
Iron, Fe^{3+}	Reagents accommodate high levels
Silver, Ag^+	If a turbidity remains and the precipitate turns black, silver interference is likely. Add 10 drops of saturated Potassium Chloride Solution to 75 mL of sample, followed by filtering through a fine or highly retentive filter. Use the filtered sample in the procedure.

Unlike CuVer 1 Reagent, CuVer 2 Reagent reacts directly with copper which is complexed by chelants such as EDTA. If free copper is to be determined separately from complexed copper, see the Powder Pillow Interference section above.

Summary of Method

Copper in the sample reacts with a salt of bicinchoninic acid contained in CuVer 1 or 2 Copper Reagent to form a purple colored complex in proportion to the copper concentration. This method includes procedures for both powder pillow and AccuVac reagents.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
CuVer 1 Copper Reagent Powder Pillows	1 pillow	100/pkg	21058-69
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

CuVer 2 Copper Reagent AccuVac Ampuls	1 ampul	25/pkg	25040-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Copper Standard Solution, 100 mg/L		100 mL	128-42
Copper Standard Solution, Voluette Ampule, 75 mg/L Cu, 10 mL		16/pkg	14247-10
CuVer 2 Reagent Powder Pillows		100/pkg	21882-99
Formaldehyde, 37%, ACS		100 mL* MDB	2059-32
Free Copper Reagent Powder Pillows		100/pkg	21186-69
Hydrochloric Acid Solution, 6.0 N		500 mL	884-49
Hydrosulfite Reagent Powder Pillows		100/pkg	21188-69
Metals Drinking Water Standard, LR for Cu, Fe, Mn		500 mL	28337-49
Metals Drinking Water Standard, HR for Cu, Fe, Mn		500 mL	28336-49
Nitric Acid, ACS		500 mL	152-49
Nitric Acid Solution, 1:1		500 mL	2540-49
Potassium Chloride Solution, saturated		100 mL	765-42
Potassium Hydroxide Standard Solution, 8.0 N		100 mL* MDB	282-32H
Sodium Hydroxide Standard Solution, 5.0 N		100 mL* MDB	2450-32
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Cylinder, graduated, mixing, 25 mL	each	20886-40
Cylinder, graduated, polypropylene, 25 mL	each	1081-40
Cylinder, graduated, 100 mL	each	508-42
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Filter Pump	each	2131-00
Flask, volumetric, 100 mL, Class A	each	14547-42
Funnel, polypropylene, 65 mm	each	1083-67
Hot Plate, 4" diameter, 120 V	each	12067-01
Hot Plate, 4" diameter, 240 V	each	12067-02
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, sens***ion 1***, with electrode	each	51700-10
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01

* Contact Hach for larger sizes.

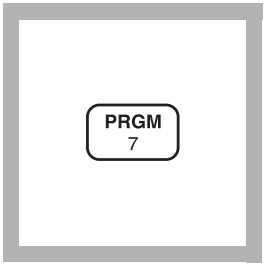
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

Porphyrin Method*

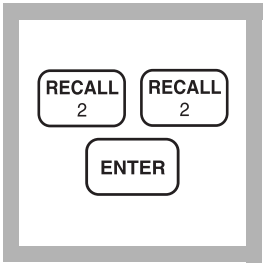


1. Enter the stored program number for copper (Cu), porphyrin method.

Press: **PRGM**

The display will show:

PRGM ?



2. Press: **22 ENTER**

The display will show **µg/L, Cu** and the **ZERO** icon**.**

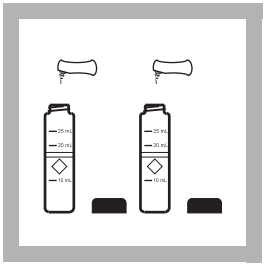
Note: *Total copper determination needs a prior digestion; use either the Digesdahl or vigorous digestion (Section 2).*

3. Fill two sample cells with 10 mL of sample.

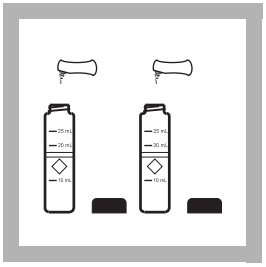
Note: *Wash all glassware with detergent. Rinse with tap water. Rinse again with Nitric Acid Solution, 1:1. Rinse a third time with copper-free, deionized water.*

4. Add the contents of one Copper Masking Reagent Powder Pillow to one of the sample cells (the blank). Swirl to dissolve.

Note: *The other sample cell is the prepared sample.*

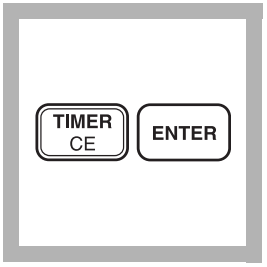


5. Add the contents of one Porphyrin 1 Reagent Powder Pillow to each sample cell. Swirl to dissolve the powder.



6. Add the contents of one Porphyrin 2 Reagent Powder Pillow to each sample cell. Swirl to dissolve the powder.

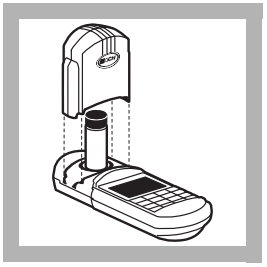
Note: *The yellow color will turn blue momentarily. If any copper is present, the yellow color will return.*



7. Press:

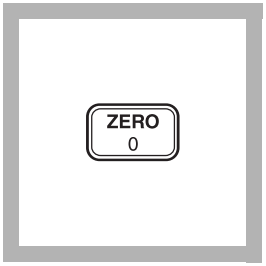
TIMER ENTER

A three-minute reaction period will begin.



8. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

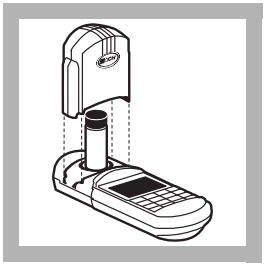
* Adapted from Ishii and Koh, *Bunseki Kagaku*, 28 473 (1979)



9. Press: **ZERO**

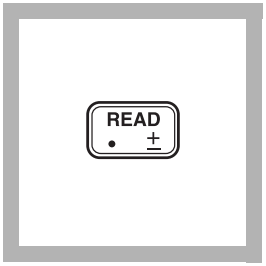
The cursor will move to the right, then the display will show:

0.0 µg/L Cu



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *If samples with high levels of metal are analyzed, a slight metallic deposit or yellow buildup may appear on the sample cell wall. Remove by rinsing with nitric acid. Dilute a fresh sample and repeat the test. Multiply the result by the dilution factor; see Section 1.*



11. Press: **READ** .

The cursor will move to the right, then the result in µg/L copper (Cu) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in acid-washed plastic bottles. To preserve, adjust the pH to 2 or less with nitric acid (about 5 mL per liter). Store preserved samples up to six months at room temperature.

Before testing, adjust the pH of the sample to between 2 and 6. If the sample is too acidic, adjust the pH with 5.0 N Sodium Hydroxide Standard Solution. Correct test results for volume additions; see *Correction for Volume Additions* in *Section 1* for more information.

Accuracy Check

Standard Additions Method

a) Fill six (3 pairs) 25-mL graduated mixing cylinders with 25 mL of sample. Properly mark each pair of cylinders as "sample" and "blank".

b) Using a TenSette Pipet, add 0.1 mL of Copper Standard Solution, 10.0 mg/L Cu, to two of the cylinders. Add 0.2 mL of standard to two more of the cylinders. Add 0.3 mL of standard to the other two cylinders, making a total of six samples (2 for each volume of standard).

c) Analyze the samples as described above. The copper concentration reading should increase by 40 µg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To assure the accuracy of the test, prepare a 100 µg/L copper standard:

a) Pipet 1.00 mL of Copper Standard Solution, 10.0 mg/L Cu, into a 100-mL volumetric flask.

b) Dilute to volume with copper-free, reagent-grade water.

c) Use this standard in place of the sample in the procedure. The reading should be 100 µg/L Cu.

d) Prepare this solution daily.

Method Performance

Precision

In a single laboratory, using a standard solution of 100 µg/L copper and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±3.4 µg/L copper.

Estimated Detection Limit

The estimated detection limit for program 22 is 5.4 µg/L Cu. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Substance	Concentration	Substance	Concentration
Aluminum	60 mg/L	Magnesium	10,000 mg/L
Cadmium	10 mg/L	Manganese	140 mg/L
Calcium	15,000 mg/L	Mercury	3 mg/L
Chloride	90,000 mg/L	Molybdenum	11 mg/L
Chromium (Cr^{6+})	110 mg/L	Nickel	60 mg/L
Cobalt	100 mg/L	Potassium	60,000 mg/L
Fluoride	30,000 mg/L	Sodium	90,000 mg/L
Iron (Fe^{2+})	6 mg/L	Zinc	9 mg/L
Lead	3 mg/L		

Chelating agents, such as EDTA, interfere at all levels unless either the Digesdahl or vigorous digestion (*Section 2*) is performed.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment: see pH Interferences in *Section 1*.

Summary of Method

The porphyrin method is very sensitive to trace amounts of free copper. Due to the sensitivity of the method, a masking agent is used to prepare a “blank” for each sample. The method is free from most interferences and does not require any sample extraction or preconcentration. Interferences from other metals are eliminated by the copper masking reagent. The porphyrin indicator forms an intense, yellow-colored complex proportional to any free copper present in the sample. Total copper may be determined if a digestion is performed prior to analysis.

REQUIRED REAGENTS

	Cat. No.
Copper Reagent Set, 10-mL samples (100 tests) Includes: (1) 26034-49, (2) 26035-49, (2) 26036-49	26033-00

Description	Quantity Required Per Test	Unit	Cat. No.
Copper Masking Reagent Powder Pillows	1 pillow	100/pkg	26034-49
Porphyrin 1 Reagent Powder Pillows	2 pillows	100/pkg	26035-49
Porphyrin 2 Reagent Powder Pillows	2 pillows	100/pkg	26036-49

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ caps	2	6/pkg	24019-06

OPTIONAL REAGENTS

Copper Standard Solution, 10 mg/L Cu	100 mL	128-42
Hydrochloric Acid Solution, 1:1 (6 N)	500 mL	884-49
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5 N	1 L	2450-53
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Beaker, 100 mL	each	500-42H
Cylinder, mixing, graduated, 25 mL	each	20886-40
Flask, volumetric, Class A, 100 mL	each	14574-42
Hot Plate, 7 x 7 inches, 120 V	each	23441-00
Hot Plate, 7 x 7 inches, 240 V	each	23441-02
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable, with electrode	each	51700-10
Pipet, Mohr, 5 mL	each	20934-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet, volumetric, 1.0 mL, Class A	each	14515-35
Pipet Filler, safety bulb	each	14651-00
Watch Glass, Pyrex®, 100 mL	each	578-70

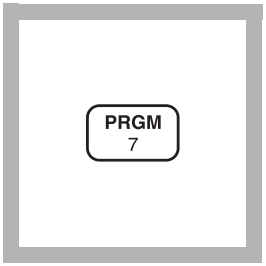
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

CYANIDE (0 to 0.240 mg/L)

Pyridine-Pyrazalone Method*



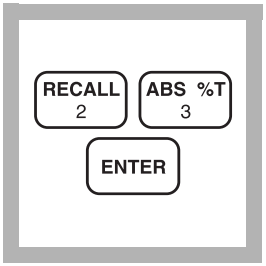
1. Enter the stored program number for cyanide (CN).

Press: **PRGM**

The display will show:

PRGM ?

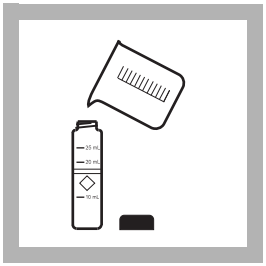
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **23 ENTER**

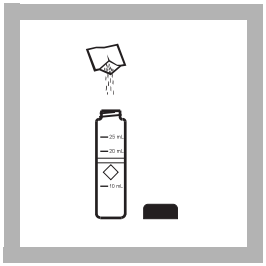
The display will show **mg/L, CN** and the **ZERO** icon**.**

Note: *Adjust the pH of stored samples before analysis.*



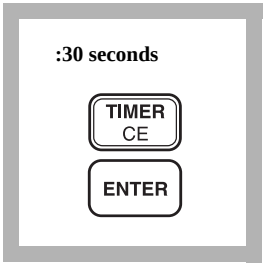
3. Fill a sample cell with 10-mL of sample.

Note: *Samples at less than 23 °C require a longer reaction time and samples at greater than 25 °C give low test results. Sample temperature must be 23-25 °C.*



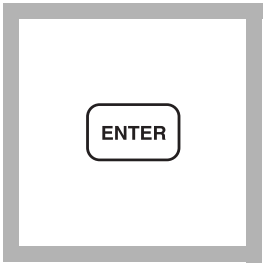
4. Add the contents of one CyaniVer 3 Cyanide Reagent Powder Pillow. Cap the sample cell.

* Adapted from Epstein, Joseph, *Anal. Chem*. 19 (4), 272 (1947)



5. Press: **TIMER ENTER**

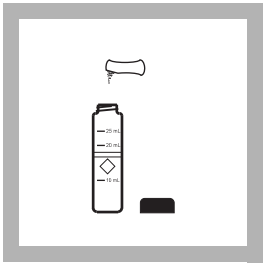
A 30-second reaction period will begin. Shake the sample cell for the 30 seconds.



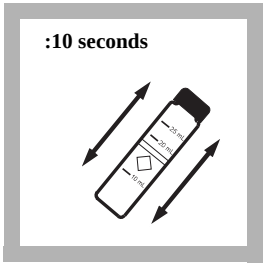
6. After the first timer beeps, the display will show: **0:30 TIMER 2**

Press **ENTER.**

A 30-second reaction period will begin. Let the sample cell sit undisturbed for this 30-second period.



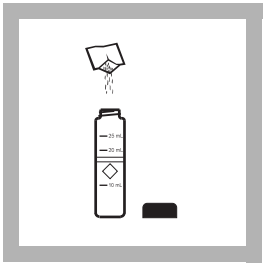
7. After the timer beeps, add the contents of one CyaniVer 4 Cyanide Reagent Powder Pillow. Cap the sample cell.



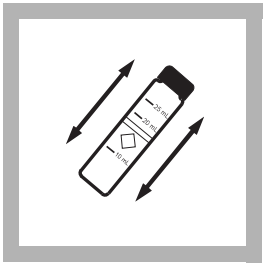
8. Shake the sample cell for ten seconds. Immediately proceed with Step 9.

Note: *Delaying the addition of the CyaniVer 5 Cyanide Reagent Powder for more than 30 seconds after the addition of the CyaniVer 4 Cyanide Reagent Powder will give lower test results.*

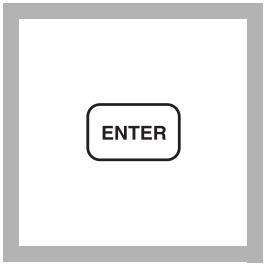
Note: *Accuracy is not affected by undissolved CyaniVer 4 Cyanide Reagent Powder.*



9. Add the contents of one CyaniVer 5 Cyanide Reagent Powder Pillow. Cap the cell.



10. Shake vigorously to completely dissolve the CyaniVer 5 Cyanide Reagent Powder (the prepared sample).



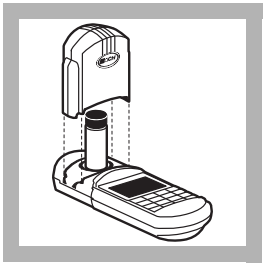
11. The display will show: **30:00 Timer 3**

Press: **ENTER**

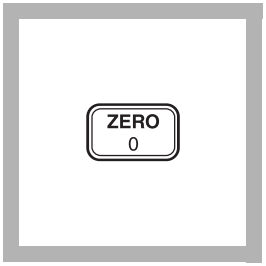
A 30-minute reaction period will begin.

Note: *If cyanide is present, a pink color will develop which then turns blue after a few minutes.*

12. Fill another 10-mL sample cell (the blank) with 10 mL of sample.



13. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

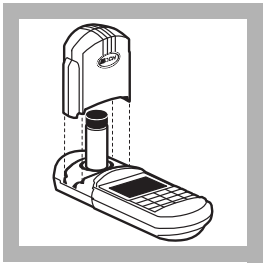


14. Press: **ZERO**

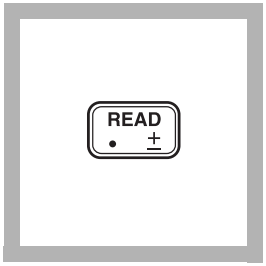
The cursor will move to the right, then the display will show:

0.000 mg/L CN

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



15. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



16. Press: **READ**

The cursor will move to the right, then the result in mg/L cyanide (CN) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in glass or plastic bottles and analyze as soon as possible.

The presence of oxidizing agents, sulfides and fatty acids can cause cyanide loss during sample storage. Samples containing these substances must be pretreated as described in the following procedures before preservation with sodium hydroxide. If the sample contains sulfide and is not pretreated, it must be analyzed within 24 hours.

Preserve the sample by adding 4.0 mL of 5.0 N Sodium Hydroxide Standard Solution to each liter (or quart) of sample, using a glass serological pipet and pipet filler. Check the sample pH. Four mL of sodium hydroxide are usually enough to raise the pH of most water and wastewater samples to 12. Add more 5.0 N sodium hydroxide if necessary. Store the samples at 4 °C (39 °F) or less. Samples preserved in this manner can be stored for 14 days.

Before testing, samples preserved with 5.0 N sodium hydroxide or samples that are highly alkaline due to chlorination treatment processes or distillation procedures should be adjusted to approximately pH 7 with 2.5 N Hydrochloric Acid Standard

Solution. If significant amounts of preservative are used, correct for the volume added; see *Correction for Volume Additions* in *Section 1* for more information.

Oxidizing Agents

Oxidizing agents such as chlorine decompose cyanides during storage. To test for their presence and eliminate their effect, pretreat the sample as follows:

a) Take a 25-mL portion of the sample and add one drop of m-Nitrophenol Indicator Solution, 10 g/L. Swirl to mix.

b) Add 2.5 N Hydrochloric Acid Standard Solution dropwise until the color changes from yellow to colorless. Swirl the sample thoroughly after the addition of each drop.

c) Add two drops of Potassium Iodide Solution, 30 g/L, and two drops of Starch Indicator Solution, to the sample. Swirl to mix. The solution will turn blue if oxidizing agents are present.

d) If Step c suggests the presence of oxidizing agents, add two level 1-g measuring spoonfuls of ascorbic acid per liter of sample.

e) Withdraw a 25-mL portion of sample treated with ascorbic acid and repeat Steps a to c. If the sample turns blue, repeat Steps d and e.

f) If the 25-mL sample remains colorless, adjust the remaining sample to pH 12 for storage with 5 N Sodium Hydroxide Standard Solution (usually 4 mL/L).

g) Perform the procedure given under Interferences, Reducing Agents, to eliminate the effect of excess ascorbic acid, before following the cyanide procedure.

Sulfides

Sulfides quickly convert cyanide to thiocyanate (SCN). To test for the presence of sulfide and eliminate its effect, pretreat the sample as follows:

a) Place a drop of sample on a disc of hydrogen sulfide test paper that has been wetted with pH 4 Buffer Solution.

b) If the test paper darkens, add a 1-g measuring spoon of lead acetate to the sample. Repeat Step a. (Purchase lead acetate from a local supplier.)

c) If the test paper continues to turn dark, keep adding lead acetate until the sample tests negative for sulfide.

d) Filter the black lead sulfide precipitate using the apparatus listed under Optional Apparatus. Preserve the sample for storage with 5 N Sodium Hydroxide Standard Solution or neutralize to a pH of 7 for analysis.

Fatty Acids

Caution—perform this operation in a hood as quickly as possible.

When distilled, fatty acids will pass over with cyanide and form soaps under the alkaline conditions of the absorber. If the presence of fatty acid is suspected, do not preserve samples with sodium hydroxide until the following pretreatment is performed. The effect of fatty acids can be minimized as follows:

a) Acidify 500 mL of sample to pH 6 or 7 with Acetic Acid Solution. (Prepare a 1:10 dilution of Acetate Acid concentration in water.)

b) Pour the sample into a 1000-mL separatory funnel and add 50 mL of hexane.

c) Stopper the funnel and shake for one minute. Allow the layers to separate.

d) Drain off the sample (lower) layer into a 600-mL beaker. If the sample is to be stored, add 5 N Sodium Hydroxide Standard Solution to raise the pH to above 12.

Accuracy Check

Standard Solution Method

Caution—Cyanides and their solutions, and the hydrogen cyanide liberated by acids, are very poisonous. Both the solutions and the gas can be absorbed through the skin.

Prepare a 100 mg/L cyanide stock solution weekly by dissolving 0.2503 grams of potassium cyanide in deionized water and diluting to 1000 mL.

Immediately before use, prepare a 0.10 mg/L cyanide working solution by diluting 1.00 mL of the 100 mg/L stock solution to 1000 mL using deionized water. Use this prepared standard in place of sample in Step 3. Results should be 0.10 mg/L CN^-.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.19 mg/L CN^- and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.017 mg/L CN^-.

Estimated Detection Limit (EDL)

The estimated detection limit for program 23 is 0.008 mg/L CN. For more information on the estimated detection limit, see *Section 1*.

Interferences

Turbidity

Large amounts of turbidity will interfere and cause high readings. If the water sample is highly turbid, it should first be filtered before use in Steps 3 and 12. Filter using the labware listed under Optional Apparatus. The test results should then be recorded as soluble cyanide.

Oxidizing and Reducing Agents

Large amounts of chlorine in the sample will cause a milky white precipitate after the addition of the CyaniVer 5 Reagent. If chlorine or other oxidizing agents are known to be present, or if reducing agents (such as sulfide or sulfur dioxide) are known to be present, use adequate ventilation and pretreat the sample before testing as follows:

Oxidizing Agents

a) Adjust a 25-mL portion of the alkaline sample to between pH 7 and 9 with 2.5 N Hydrochloric Acid Standard Solution. Count the number of drops of acid added.

b) Add two drops of Potassium Iodide Solution and two drops of Starch Indicator Solution to the sample. Swirl to mix. The sample will turn blue if oxidizing agents are present.

c) Add Sodium Arsenite Solution drop-wise until the sample turns colorless. Swirl the sample thoroughly after each drop. Count the number of drops.

d) Take another 25-mL sample and add the total number of drops of Hydrochloric Acid Standard Solution counted in Step a.

e) Subtract one drop from the amount of Sodium Arsenite Solution added in Step c. Add this amount to the sample and mix thoroughly.

f) Using 10 mL of this sample, continue with Step 3 of the cyanide procedure.

Reducing Agents

a) Adjust a 25-mL portion of the alkaline sample to between pH 7 and 9 with 2.5 N Hydrochloric Acid Standard Solution. Count the number of drops added.

b) Add four drops of Potassium Iodide Solution and four drops of Starch Indicator Solution to the sample. Swirl to mix. The sample should be colorless.

c) Add Bromine Water drop-wise until a blue color appears. Count the number of drops, and swirl the sample after the addition of each drop.

d) Take another 25 mL sample and add the total number of drops of Hydrochloric Acid Standard Solution counted in Step a.

e) Add the total number of drops of Bromine Water counted in Step c to the sample and mix thoroughly.

f) Using 10 mL of this sample, continue with Step 3 of the cyanide procedure.

Metals

Nickel or cobalt in concentrations up to 1 mg/L do not interfere. Eliminate the interference from up to 20 mg/L copper and 5 mg/L iron by adding the contents of one HexaVer Chelating Reagent Powder Pillow to the sample and then mixing before adding the CyaniVer 3 Cyanide Reagent Powder Pillow in Step 4. Prepare a reagent blank of deionized water and reagents to zero the instrument in Step 13.

Acid Distillation

For USEPA reporting purposes, samples must be distilled.

All samples should be treated by acid distillation except when experience has shown that there is no difference in results obtained with or without distillation. With most compounds, a one-hour reflux is adequate.

If thiocyanate is present in the original sample, a distillation step is absolutely necessary as thiocyanate causes a positive interference. High concentrations of thiocyanate can yield a substantial quantity of sulfide in the distillate. The “rotten egg” smell of hydrogen sulfide will accompany the distillate when sulfide is present. The sulfide must be removed from the distillate prior to testing.

If cyanide is not present, the amount of thiocyanate can be determined. The sample is not distilled and the final reading is multiplied by 2.2. The result is mg/L thiocyanate.

The distillate can be tested and treated for sulfide after the last step of the distillation procedure by using the following lead acetate
treatment procedure.

a) Place a drop of the distillate (already diluted to 250 mL) on a disc of hydrogen sulfide test paper that has been wetted with pH 4.0 Buffer Solution.

b) If the test paper darkens, add 2.5 N Hydrochloric Acid Standard Solution drop-wise to the distillate until a neutral pH is obtained.

c) Add a 1-g measuring spoon of lead acetate to the distillate and mix. Repeat Step a.

d) If the test paper continues to turn dark, keep adding lead acetate until the distillate tests negative for sulfide.

e) Filter the black lead sulfide precipitate through filter paper and funnel. This sample should now be neutralized to pH 7 and analyzed for cyanide without delay.

Distillation Procedures

A detailed procedure for the distillation of cyanide samples is included with the Hach Distillation Apparatus. Three detailed procedures, Free Cyanides, Cyanides Amenable to Chlorination, and Total Cyanides, are included with the four- and ten-position Midi-Dist Distillation System. See the Optional Apparatus listing.

Summary of Method

The pyridine-pyrazolone method gives an intense blue color with free cyanide. A sample distillation is required to determine cyanide from transition and heavy metal cyanide complexes.

REQUIRED REAGENTS

	Cat. No.
Cyanide Reagent Set (100 Tests), 10 mL samples	24302-00
Includes: (1) 21068-69, (1) 21069-69, (1) 21070-69	

Description	Quantity Required Per Test	Unit	Cat. No.
CyaniVer 3 Cyanide Reagent Powder Pillows	1 pillow	100/pkg	21068-69
CyaniVer 4 Cyanide Reagent Powder Pillows	1 pillow	100/pkg	21069-69
CyaniVer 5 Cyanide Reagent Powder Pillows	1 pillow	100/pkg	21070-69

REQUIRED APPARATUS

Sample Cell, 10-20-25, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Acetic Acid, Glacial	500 mL	100-49
Ascorbic Acid	100 g	6138-26
Bromine Water	25 mL	2211-20
Buffer Solution, pH 4.0	500 mL	12223-49
Hexanes, ACS	500 mL	14478-49
HexaVer Chelating Reagent Powder Pillows	100/pkg	243-99
Hydrochloric Acid Standard Solution, 2.5 N	100 mL MDB	1418-32
Magnesium Chloride Solution	1 L	14762-53
m-Nitrophenol Indicator	100 mL MDB	2476-32
Potassium Iodide Solution, 30 g/L	100 mL MDB	343-32
Sodium Arsenite Solution, APHA	100 mL MDB	1047-32
Potassium Cyanide, ACS	28 g	767-14
Sodium Hydroxide Standard Solution, 0.25 N	1 L	14763-53
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53

OPTIONAL REAGENTS (continued)

Description	Unit	Cat. No.
Starch Indicator Solution	10 mL MDB	349-32
Sulfuric Acid Standard Solution, 19.2 N	500 mL	2038-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Beaker, glass, 600 mL	each	500-52
Bottle, wash, 500 mL	each	620-11
Cylinder, graduated, 50 mL	each	508-41
Cylinder, graduated, 250 mL	each	508-46
Distillation Apparatus, cyanide accessories	each	22658-00
Distillation Apparatus, general purpose accessories	each	22653-00
Distillation Apparatus Heater and Support Apparatus, 115 Vac, 60 Hz	each	22744-00
Distillation Apparatus Heater and Support Apparatus, 230 Vac, 50 Hz	each	22744-02
Dropper, plastic	each	6080-00
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, volumetric, Class A, 1000 mL	each	14574-53
Flask, volumetric, Class A, 250 mL	each	14574-46
Funnel, poly, 65 mm	each	1083-67
Funnel, separatory, 500 mL	each	520-49
Hydrogen Sulfide Test Papers	100/pkg	25377-33
pH Meter, *sens**ion***™*1*, portable	each	51700-10
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00
Scoop, double ended	each	12257-00
Spoon, measuring, 1.0 g	each	510-00
Support Ring, 4 inch	each	580-01
Support Stand	each	563-00
Thermometer, –20 to 110 °C, non-mercury	each	26357-02

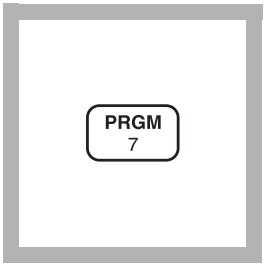
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

CYANURIC ACID (7 to 55 mg/L)

Turbidimetric Method



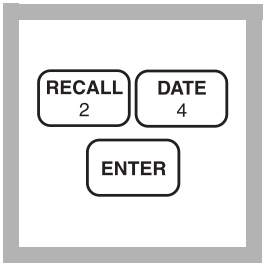
1. Enter the stored program number for cyanuric acid.

Press: **PRGM**

The display will show:

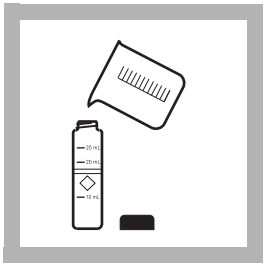
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



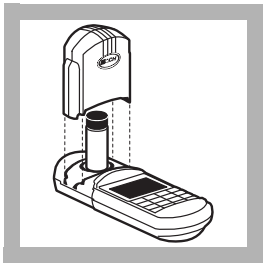
2. Press: **24 ENTER**

The display will show **mg/L, CYACD** and the **ZERO** icon.

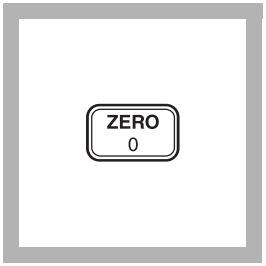


3. Fill a sample cell with 25 mL of sample (the blank).

Note: *Filtering is required for highly turbid samples.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

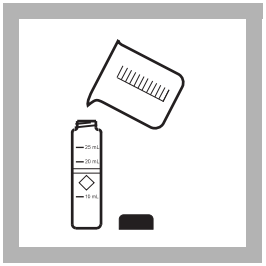


5. Press: **ZERO**

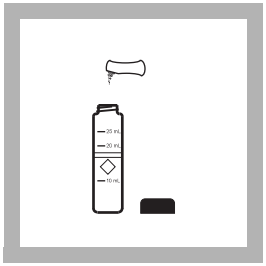
The cursor will move to the right, then the display will show:

0 mg/L CYACD

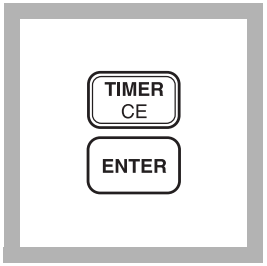
Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



6. Fill another cell with 25 mL of sample.



7. Add the contents of one Cyanuric Acid 2 Reagent Powder Pillow (the prepared sample). Swirl to mix.

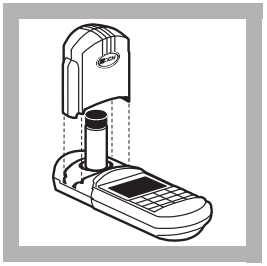


8. Press **TIMER ENTER**

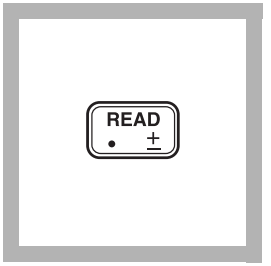
A three-minute reaction period will begin.

Note: *A white turbidity will form if cyanuric acid is present.*

Note: *Accuracy is not affected by undissolved powder.*



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L cyanuric acid will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Note: *Clean sample cells with soap, water and a brush soon after each test to prevent a white film from forming.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Samples must be analyzed within 24 hours.

Accuracy Check

Standard Solution Method

a) Dissolve 1.000 gram of cyanuric acid in 1000 mL of deionized water to make a 1000 mg/L solution. It takes several hours for the cyanuric acid to dissolve. This solution is stable for several weeks.

b) Dilute 2.00 mL of the 1000 mg/L solution to 100 mL with deionized water to make a 20 mg/L solution. Prepare fresh daily.

c) Testing the 20 mg/L solution should give test results of about 20 mg/L cyanuric acid.

Method Performance

Precision

In a single laboratory, using a standard solution of 25.0 mg/L cyanuric acid and two lots of reagent with the instrument, a single

operator obtained a standard deviation of ±1.2 mg/L cyanuric acid.

Estimated Detection Limit

The estimated detection limit for program 24 is 7.0 mg/L cyanuric acid. For more information on the estimated detection limit, see *Section 1*.

Interferences

Turbidity will interfere. Filter turbid samples before running the test.

Summary of Method

The test for cyanuric acid uses the turbidimetric method. Cyanuric Acid 2 Reagent precipitates any cyanuric acid present and holds it in suspension. The amount of turbidity caused by the suspended particles is directly proportional to the amount of cyanuric acid present. Due to the nature of the precipitation reaction, low levels of cyanuric acid (less than 7 mg/L) are not detected by this method.

REQUIRED REAGENTS AND APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Cyanuric Acid 2 Reagent Powder Pillow	1 pillow	50/pkg	2460-66
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Cyanuric Acid		25 g	7129-24
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Balance, Acculab UI Series		each	26947-00
Filter Paper, folded 12.5 cm		100/pkg	1894-57
Flask, volumetric, Class A, 100 mL		each	14574-42
Flask, volumetric, Class A, 1000 mL		each	14574-53
Funnel, poly, 65 mm		each	1083-67
Pipet, Bulb		each	14651-00
Pipet, volumetric, Class A, 2.00 mL		each	14515-36

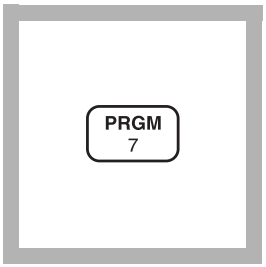
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

DEHA (0 to 500 µg/L)

Iron Reduction Method for Oxygen Scavengers*



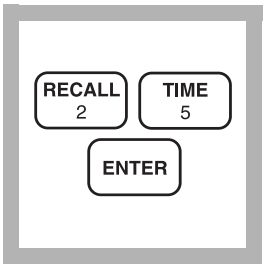
1. Enter the stored program number for diethylhydroxylamine (DEHA).

Press: **PRGM**

The display will show:

PRGM ?

Note: *To determine other oxygen scavengers, multiply the result by the appropriate factor. See* Other Oxygen Scavengers *following these steps.*

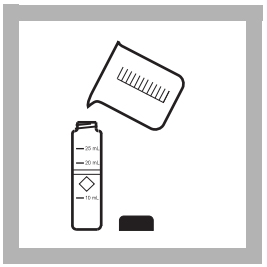


2. Press: **25 ENTER**

The display will show **µg/L, DEHA** and the **ZERO** icon**.**

Note: *To prevent contamination from iron deposits, rinse sampling containers and sample cells with 1:1 Hydrochloric Acid Solution. Follow with several rinsings of deionized water.*

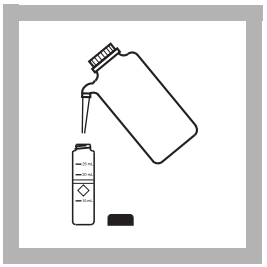
Note: *Samples must be analyzed immediately.*



3. Fill a sample cell with 25 mL of sample (the prepared sample).

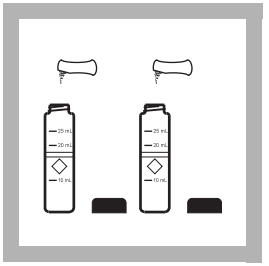
Note: *The sample temperature should be 25 ±3°C (77 ±5 °F).*

Note: *When testing for compounds that react quickly with oxygen at room temperature, stopper the cell containing the sample in Steps 5–11.*

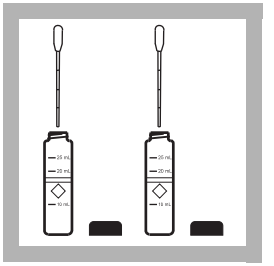


4. Fill a second sample cell with 25 mL of deionized water (the blank).

* Adapted from Ishii and Koh, *Buneseki Kagaku,* 28 473 (1979)

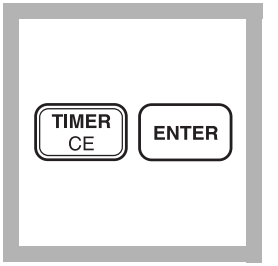


5. Add the contents of one DEHA Reagent 1 Powder Pillow to each sample cell. Cap. Swirl to mix.



6. Add exactly 0.5 mL of DEHA Reagent 2 Solution to each sample cell. Cap and swirl to mix. Place both sample cells in the dark.

Note: *A purple color will slowly develop if DEHA is present.*



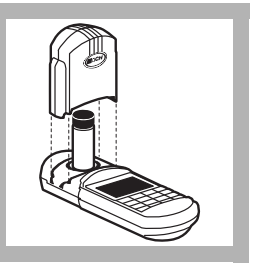
7. Immediately, press:

TIMER ENTER

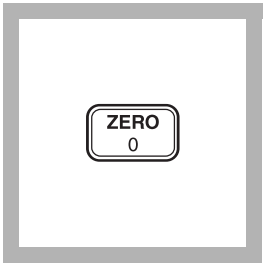
A 10-minute reaction period will begin. For hydroquinone, allow only a two-minute reaction period.

Note: *Both sample cells must remain in the dark for the entire reaction period.*

Note: *Temperature and reaction time affect results.*



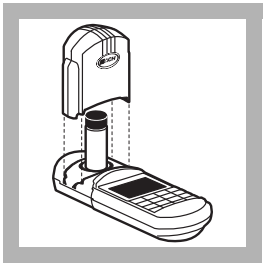
8. Immediately after the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



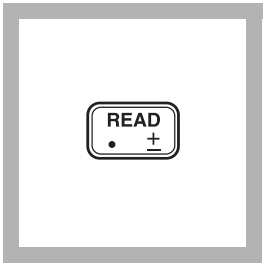
9. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 µg/L DEHA



10. Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in µg/L DEHA will be displayed.

Note: *If the display flashes "limit" it is due to high DEHA levels. Dilute a fresh sample with deoxygenated deionized water and repeat the test. Multiply the result by the dilution factor; see Section 1.*

Ferrous Iron Adjustment

Note: *Repeat the above procedure, but do not add DEHA Reagent 2 (Step 6) to determine the ferrous iron content in the sample. Then press SETUP, scroll to "BLANK" and press* **ENTER**. *The display will show; "BLANK?" Enter the blank value just read. Press* **ENTER** *to accept the value as the blank to be subtracted from each reading.*

Sampling and Storage

Most oxygen scavengers will react quickly with atmospheric oxygen. Collect samples in acid-rinsed plastic or glass containers, allowing the sample to overflow. Cap the container so there is no head space above the sample. Rinse each sample cell several times with sample, then carefully fill to the fill mark. Analyze the sample immediately.

Other Oxygen Scavengers

To determine other oxygen scavengers, perform the test as directed above, then multiply the DEHA result by the appropriate factor below:

Oxygen Scavenger	Factor
Erythorbic Acid (Iso-ascorbic acid)	3.5
Hydroquinone	2.5
Methylethylketoxime (MEKO)	4.1
Carbohydrazide	1.3

Method Performance

Precision

In a single laboratory, using a standard solution of 242 µg/L DEHA and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±6.2 µg/L DEHA.

Estimated Detection Limit

The estimated detection limit for program 25 is 9 µg/L DEHA. For more information on the estimated detection limit, see *Section 1*.

Interferences

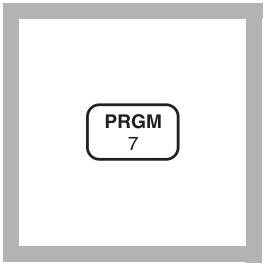
Substances which reduce ferric iron will interfere. Substances which complex iron strongly may also interfere. Light interferes with the color development. The following may also interfere when present in concentrations exceeding those listed below:

Borate (as $Na_2B_4O_7$)	500 mg/L	Molybdenum	80 mg/L
Cobalt	0.025 mg/L	Nickel	0.8 mg/L
Copper	8.0 mg/L	Phosphate	10 mg/L
Hardness (as $CaCO_3$)	1000 mg/L	Phosphonates	10 mg/L
Lignosulfonates	0.05 mg/L	Sulfate	1000 mg/L
Manganese	0.8 mg/L	Zinc	50 mg/L

FLUORIDE (0 to 2.00 mg/L F^-)

SPADNS Method* (Reagent Solution or AccuVac Ampuls)

Using SPADNS Reagent Solution

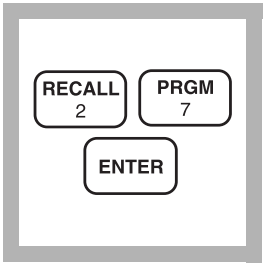


1. Enter the stored program number for fluoride (F) powder pillows.

Press: **PRGM**

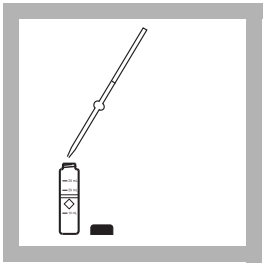
The display will show:

PRGM ?

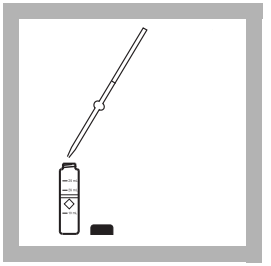


2. Press: **27 ENTER**

The display will show **mg/L, F** and the **ZERO** icon.

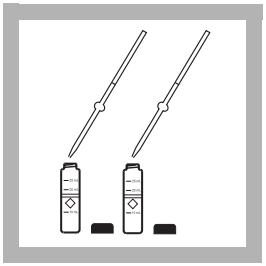


3. Pipet 10.0 mL of sample into a dry 10-mL sample cell (the prepared sample).



4. Measure 10.0 mL of deionized water into a second dry sample cell (the blank).

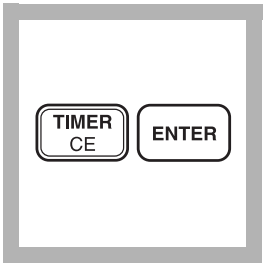
Note: *The sample and blank should be at the same temperature (±1 °C). Temperature adjustments may be made before or after reagent addition.*



5. Pipet 2.00 mL of SPADNS Reagent into each cell. Swirl to mix.

Note: *SPADNS Reagent is toxic and corrosive; use care while measuring. Use a pipet filler.*

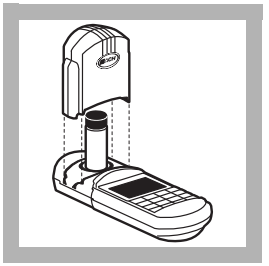
Note: *The SPADNS Reagent must be measured accurately.*



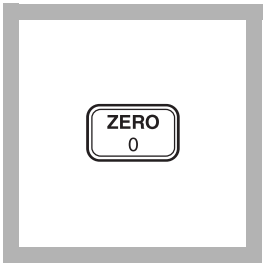
6. Press:

TIMER ENTER

A one minute reaction period will begin.



7. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L F

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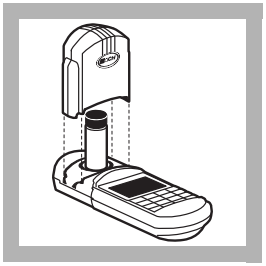
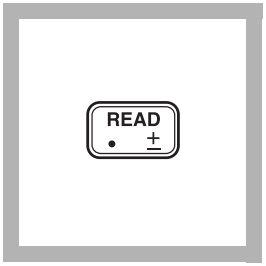
* Adapted from *Standard Methods for the Examination of Water and Wastewater. The procedure for this instrument uses an alternate wavelength outside the accepted 550-580 nm range. The reagents used are the same as those in the USEPA accepted method.*



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

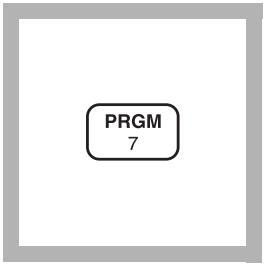


10. Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

Using AccuVac Ampuls

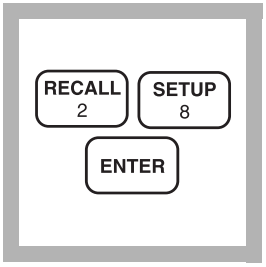


1. Enter the stored program number for fluoride (F^-)- AccuVac Ampuls.

Press: **PRGM**

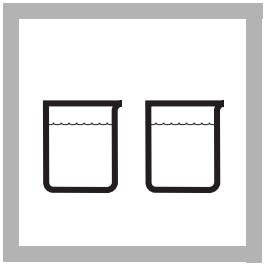
The display will show:

PRGM ?

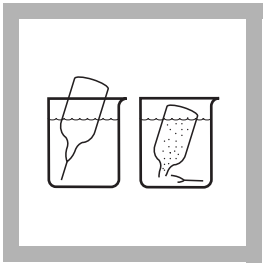


2. Press: **28 ENTER**

The display will show **mg/L, F** and the **ZERO** icon**.**

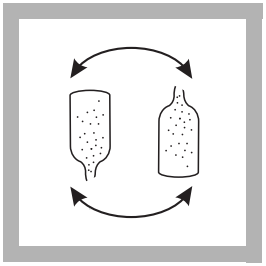


3. Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second beaker.



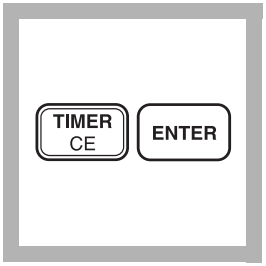
4. Fill a SPADNS Fluoride Reagent AccuVac Ampul with sample by breaking the tip on the bottom of the beaker. Fill a second AccuVac Ampul with deionized water (the blank).

Note: *Keep the tip immersed while the ampule fills completely.*



5. Quickly invert the ampules several times to mix. Wipe off any liquid or fingerprints.

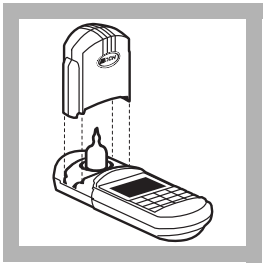
Note: *Do not place finger over the broken tip- the liquid will remain in the ampul.*



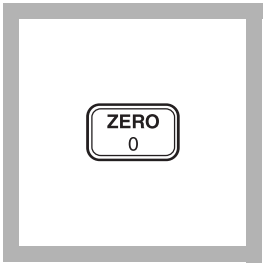
6. Press:

TIMER ENTER

A one-minute reaction period will begin.



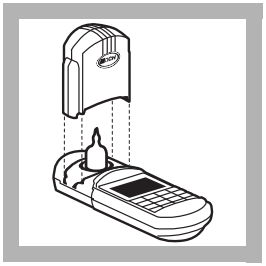
7. After the timer beeps place the blank into the cell holder. Tightly cover the ampule with the instrument cap.



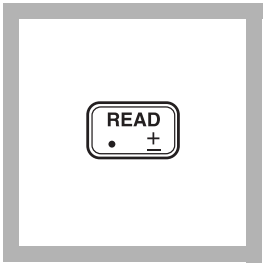
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L F



9. Place the AccuVac Ampul containing the sample into the instrument. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

Sampling and Storage

Collect samples in plastic bottles. Samples may be stored up to 28 days.

Accuracy Check

Standard Solution Method

A variety of standard solutions covering the entire range of the test are available from Hach. Use these in place of sample to verify technique. Minor variations between lots of reagent become measurable above
1.5 mg/L. While results in this region are usable for most purposes, better accuracy may be obtained by diluting a fresh sample 1:1 with deionized water and retesting. Multiply the result by 2.

Standard Adjust

To adjust the calibration curve using the reading obtained with a 1.80-mg/L Standard Solution, press **SETUP** and use the arrow keys to scroll to the "STD" setup option. Press **ENTER** to activate the option. Then enter **1.80** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment* in *Section 1* for more information.

Method Performance

Precision

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS Reagent with the instrument, a single operator obtained standard deviations of ±0.035 mg/L fluoride.

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS AccuVac Reagent with the instrument, a single operator obtained standard deviations of ±0.040 mg/L fluoride.

Estimated Detection Limit (EDL)

The EDL for programs 27 and 28 is 0.05 mg/L F^-. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

This test is sensitive to small amounts of interference. Glassware must be very clean. Repeating the test with the same glassware is recommended to ensure that results are accurate.

The following substances interfere to the extent shown:

Substance	Concentration	Error
Alkalinity (as $CaCO_3$)	5000 mg/L	-0.1 mg/L F^-
Aluminum	0.1 mg/L	-0.1 mg/L F^-
Chloride	7000 mg/L	+0.1 mg/L F^-
Iron, ferric	10 mg/L	-0.1 mg/L F^-
Phosphate, ortho	16 mg/L	+0.1 mg/L F^-
Sodium Hexametaphosphate	1.0 mg/L	+0.1 mg/L F^-
Sulfate	200 mg/L	+0.1 mg/L F^-

SPADNS Reagent contains enough arsenite to eliminate interference up to 5 mg/L chlorine. For higher chlorine levels, add one drop of Sodium Arsenite Solution to 25 mL of sample for each 2 mg/L of chlorine.

To check for interferences from aluminum, read the concentration one minute after reagent addition, then again after 15 minutes. An appreciable increase in concentration suggests aluminum interference. Waiting two hours before making the final reading will eliminate the effect of up to 3.0 mg/L aluminum.

Most interferences can be eliminated by distilling the sample from an acid solution as described below:

a) Set up the distillation apparatus for the general purpose distillation. See the Hach Distillation Apparatus Manual. Turn on the water and make certain it is flowing through the condenser.

b) Measure 100 mL of sample into the distillation flask. Add a magnetic stirring bar and turn on the heater power switch.
Turn the stir control to 5.

c) Cautiously measure 150 mL of StillVer Distillation Solution (2:1 Sulfuric Acid) into the flask. If high levels of chloride are present, add 5 mg silver sulfate for each mg/L chloride present.

d) Turn the heat control to setting 10, with the thermometer in place. The yellow pilot lamp shows when the heater is on.

e) When the temperature reaches 180 °C (about one hour), turn the still off.

f) Dilute the collected distillate to 100 mL, if necessary. Analyze the distillate by the above method.

Summary of Method

The SPADNS Method for fluoride determination involves the reaction of fluoride with a red zirconium-dye solution. The fluoride combines with part of the zirconium to form a colorless complex, thus bleaching the red color in an amount proportional to the fluoride concentration. Seawater and wastewater samples require distillation. See Optional Apparatus for Distillation Apparatus listing.

Pollution Prevention and Waste Management

SPADNS Reagent contains sodium arsenite. Final solutions will contain sodium arsenite (D004) in sufficient concentration to be regulated as hazardous waste for Federal RCRA. See *Section 3* for more information on disposal of these materials.

REQUIRED REAGENTS (Using Solution)

Description	Quantity Required Per Test	Unit	Cat. No.
SPADNS Reagent for Fluoride	4 mL	500 mL	444-49
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS (Using Solution)

Pipet Filler safety bulb	1	each	14651-00
Pipet, volumetric, Class A, 10.00 mL	1	each	14515-38
Pipet, volumetric, Class A, 2.00 mL	1	each	14515-36
Sample Cell, 10-20-25 mL w/ cap	2	6/pkg	24019-06
Thermometer, –20 to 110°C, non-mercury	1	each	26357-02

REQUIRED REAGENTS (Using AccuVac Ampuls)

SPADNS Fluoride Reagent AccuVac Ampuls	2 ampuls	25/pkg	25060-25
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	2	each	500-41H

OPTIONAL REAGENTS

Drinking Water Inorganics Standard for F^-, NO_3^-, PO_4^{3-}, and SO_4^{2-}	500 mL		28330-49
Fluoride Standard Solution, 0.2 mg/L F^-	500 mL		405-02
Fluoride Standard Solution, 0.5 mg/L F^-	500 mL		405-05
Fluoride Standard Solution, 0.8 mg/L F^-	500 mL		405-08
Fluoride Standard Solution, 1.0 mg/L F^-	1000 mL		291-53
Fluoride Standard Solution, 1.0 mg/L F^-	500 mL		291-49
Fluoride Standard Solution, 1.2 mg/L F^-	500 mL		405-12
Fluoride Standard Solution, 1.5 mg/L F^-	500 mL		405-15
Fluoride Standard Solution, 2.0 mg/L F^-	500 mL		405-20
Silver Sulfate, ACS	113 g		334-14
Sodium Arsenite Solution	100 mL MDB		1047-32
StillVer Distillation Solution	500 mL		446-49

OPTIONAL APPARATUS

AccuVac Snapper Kit		each	24052-00
Cylinder, graduated, 100 mL		each	508-42
Cylinder, graduated, 250 mL		each	508-46
Distillation Heater and Support Apparatus Set, 115 V, 50/60 Hz		each	22744-00
Distillation Heater and Support Apparatus Set, 230 V, 50/60 Hz		each	22744-02
Distillation Apparatus General Purpose Accessories		each	22653-00
pH Meter, *sens**ion***™***1,** portable, with electrode		each	51700-10
Pipet, TenSette, 1.0 to 10.0 mL		each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet		50/pkg	21997-96
Stopper		6/pkg	1731-06

For Technical Assistance, Price and Ordering

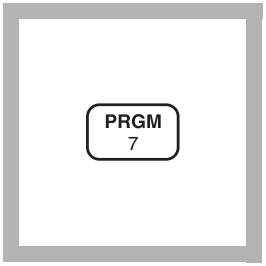
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

FLUORIDE (0 to 2.00 mg/L F^-)

SPADNS 2 Method* (Reagent Solution or AccuVac Ampuls)

Using SPADNS 2 Reagent Solution

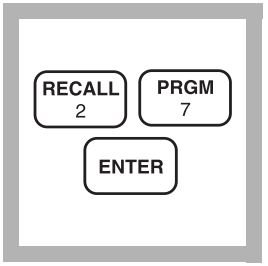


1. Enter the stored program number for fluoride (F^-) powder pillows.

Press: **PRGM**

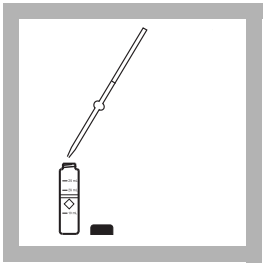
The display will show:

PRGM ?

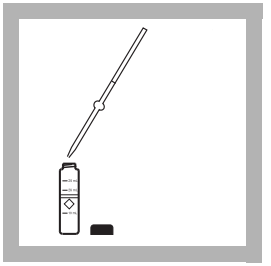


2. Press: **27 ENTER**

The display will show **mg/L, F** and the **ZERO** icon.

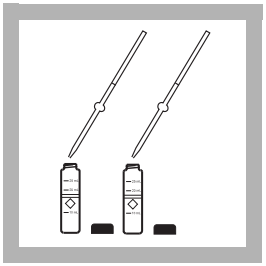


3. Pipet 10.0 mL of sample into a dry 10-mL sample cell (the prepared sample).



4. Measure 10.0 mL of deionized water into a second dry sample cell (the blank).

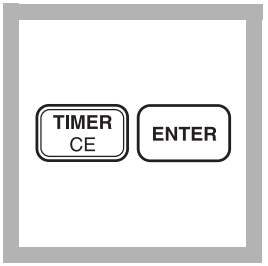
Note: *The sample and blank should be at the same temperature (±1 °C). Temperature adjustments may be made before or after reagent addition.*



5. Pipet 2.00 mL of SPADNS 2 Reagent into each cell. Swirl to mix.

Note: *SPADNS 2 Reagent is corrosive; use care while measuring. Use a pipet filler.*

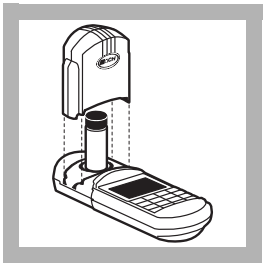
Note: *The SPADNS 2 Reagent must be measured accurately.*



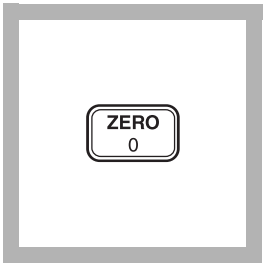
6. Press:

TIMER ENTER

A one minute reaction period will begin.



7. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



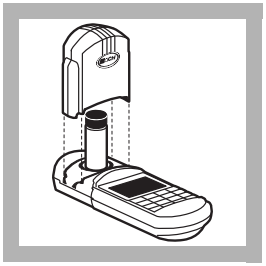
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

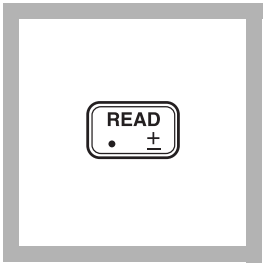
0.00 mg/L F

---

* Adapted from *Standard Methods for the Examination of Water and Wastewater. Per USEPA Rules and Regulations at 40 CFR 136.6, Method Modifications and Analytical Requirements, Hach Method 10225 (SPADNS 2) for the determination of fluoride in water is equivalent to the EPA Reference Method SM 4500-F D. Equivalency data is available upon request.*



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

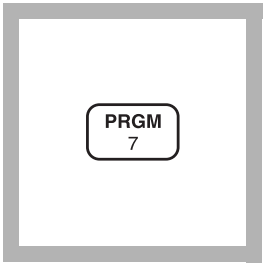


10. Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

Using AccuVac Ampuls

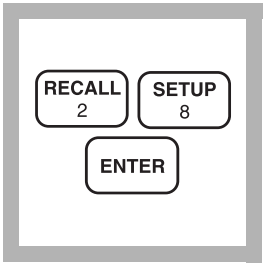


1. Enter the stored program number for fluoride (F^-) AccuVac Ampuls.

Press: **PRGM**

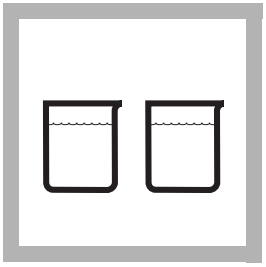
The display will show:

PRGM ?

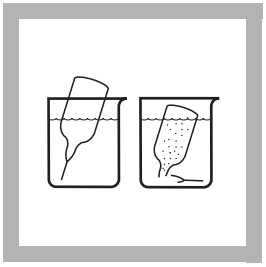


2. Press: **28 ENTER**

The display will show **mg/L, F** and the **ZERO** icon**.**

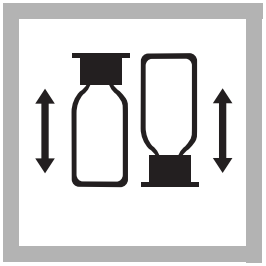


3. Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second beaker.

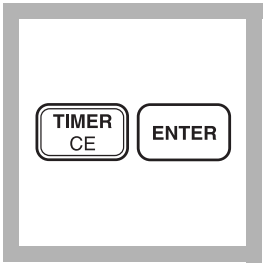


4. Fill an SPADNS 2 Fluoride Reagent AccuVac Ampul with sample by breaking the tip on the bottom of the beaker. Fill a second AccuVac Ampul with deionized water (the blank).

Note: *Keep the tip immersed while the ampule fills completely.*



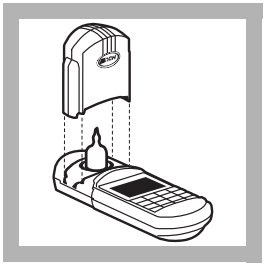
5. Cap and quickly invert the ampules several times to mix. Wipe off any liquid or fingerprints.



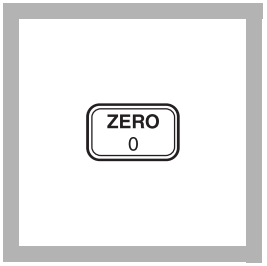
6. Press:

TIMER ENTER

A one-minute reaction period will begin.



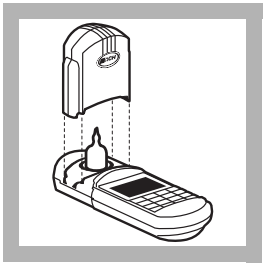
7. After the timer beeps place the blank into the cell holder. Tightly cover the ampule with the instrument cap.



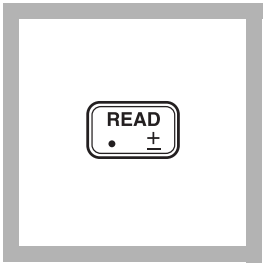
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L F



9. Place the AccuVac Ampul containing the sample into the instrument. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L fluoride will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check following these steps.*

Sampling and Storage

Collect samples in plastic bottles. Samples may be stored up to 28 days.

Accuracy Check

Standard Solution Method

A variety of standard solutions covering the entire range of the test are available from Hach. Use these in place of sample to verify technique. Minor variations between lots of reagent become measurable above
1.5 mg/L. While results in this region are usable for most purposes, better accuracy may be obtained by diluting a fresh sample 1:1 with deionized water and retesting. Multiply the result by 2.

Standard Adjust

To adjust the calibration curve using the reading obtained with a 1.80-mg/L Standard Solution, press **SETUP** and use the arrow keys to scroll to the "STD" setup option. Press **ENTER** to activate the option. Then enter **1.80** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment* in *Section 1* for more information.

Method Performance

Precision

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS 2 Reagent with the instrument, a single operator obtained standard deviations of ±0.035 mg/L fluoride.

In a single laboratory, using standard solutions of 1.00 mg/L fluoride and two lots of SPADNS 2 AccuVac Reagent with the instrument, a single operator obtained standard deviations of ±0.040 mg/L fluoride.

Estimated Detection Limit (EDL)

The EDL for programs 27 and 28 is 0.05 mg/L F^-. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

This test is sensitive to small amounts of interference. Glassware must be very clean. Repeating the test with the same glassware is recommended to ensure that results are accurate.

The following substances interfere to the extent shown:

Substance	Concentration	Error
Alkalinity (as $CaCO_3$)	5000 mg/L	-0.1 mg/L F^-
Aluminum	0.1 mg/L	-0.1 mg/L F^-
Chloride	7000 mg/L	+0.1 mg/L F^-
Iron, ferric	10 mg/L	-0.1 mg/L F^-
Phosphate, ortho	16 mg/L	+0.1 mg/L F^-
Sodium Hexametaphosphate	1.0 mg/L	+0.1 mg/L F^-
Sulfate	200 mg/L	+0.1 mg/L F^-

SPADNS 2 Reagent contains enough non-toxic reducing agent to eliminate interference up to 5 mg/L chlorine. For higher chlorine levels, dilute sample with deionized water by a factor that will lower chlorine concentration to below 5 mg/L. Perform the procedure, and multiply results by this factor to obtain mg/L Fluoride.

To check for interferences from aluminum, read the concentration one minute after reagent addition, then again after 15 minutes. An appreciable increase in concentration suggests aluminum interference. Waiting two hours before making the final reading will eliminate the effect of up to 3.0 mg/L aluminum.

Most interferences can be eliminated by distilling the sample from an acid solution as described below:

a) Set up the distillation apparatus for the general purpose distillation. See the Hach Distillation Apparatus Manual. Turn on the water and make certain it is flowing through the condenser.

b) Measure 100 mL of sample into the distillation flask. Add a magnetic stirring bar and turn on the heater power switch.
Turn the stir control to 5.

c) Cautiously measure 150 mL of StillVer Distillation Solution (2:1 Sulfuric Acid) into the flask. If high levels of chloride are present, add 5 mg silver sulfate for each

mg/L chloride present.

d) Turn the heat control to setting 10, with the thermometer in place. The yellow pilot lamp shows when the heater is on.

e) When the temperature reaches 180 °C (about one hour), turn the still off.

f) Dilute the collected distillate to 100 mL, if necessary. Analyze the distillate by the above method.

Summary of Method

The SPADNS 2 Method for fluoride determination involves the reaction of fluoride with a red zirconium-dye solution. The fluoride combines with part of the zirconium to form a colorless complex, thus bleaching the red color in an amount proportional to the fluoride concentration. Seawater and wastewater samples require distillation. See Optional Apparatus for Distillation Apparatus listing.

Pollution Prevention and Waste Management

SPADNS 2 Reagent contains a non-toxic proprietary reducing agent in place of sodium arsenite.

REQUIRED REAGENTS (Using Solution)

Description	Quantity Required Per Test	Unit	Cat. No.
SPADNS 2 Reagent for Fluoride	4 mL	500 mL	29475-49
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS (Using Solution)

Pipet Filler safety bulb	1	each	14651-00
Pipet, volumetric, Class A, 10.00 mL	1	each	14515-38
Pipet, volumetric, Class A, 2.00 mL	1	each	14515-36
Sample Cell, 10-20-25 mL w/ cap	2	6/pkg	24019-06
Thermometer, –20 to 110°C, non-mercury	1	each	26357-02

REQUIRED REAGENTS (Using AccuVac Ampuls)

SPADNS 2 Fluoride Reagent AccuVac Ampuls	2 ampuls	25/pkg	25270-25
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	2	each	500-41H

OPTIONAL REAGENTS

Drinking Water Inorganics Standard for F^-, NO_3^-, PO_4^{3-}, and SO_4^{2-}	500 mL	28330-49
Fluoride Standard Solution, 0.2 mg/L F^-	500 mL	405-02
Fluoride Standard Solution, 0.5 mg/L F^-	500 mL	405-05
Fluoride Standard Solution, 0.8 mg/L F^-	500 mL	405-08
Fluoride Standard Solution, 1.0 mg/L F^-	1000 mL	291-53
Fluoride Standard Solution, 1.0 mg/L F^-	500 mL	291-49
Fluoride Standard Solution, 1.2 mg/L F^-	500 mL	405-12
Fluoride Standard Solution, 1.5 mg/L F^-	500 mL	405-15
Fluoride Standard Solution, 2.0 mg/L F^-	500 mL	405-20
Silver Sulfate, ACS	113 g	334-14
StillVer Distillation Solution	500 mL	446-49

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Cylinder, graduated, 100 mL	each	508-42
Cylinder, graduated, 250 mL	each	508-46
Distillation Heater and Support Apparatus Set, 115 V, 50/60 Hz	each	22744-00
Distillation Heater and Support Apparatus Set, 230 V, 50/60 Hz	each	22744-02
Distillation Apparatus General Purpose Accessories	each	22653-00
pH Meter, *sens**ion***™***1,** portable, with electrode	each	51700-10
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg	21997-96
Stopper	6/pkg	1731-06

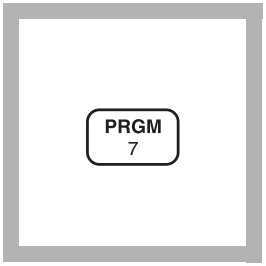
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

HARDNESS (0 to 4.00 mg/L Ca and Mg as $CaCO_3$) For water, wastewater, seawater

Calcium and Magnesium; Calmagite Colorimetric Method



1. Enter the stored program number for magnesium hardness (as $CaCO_3$).

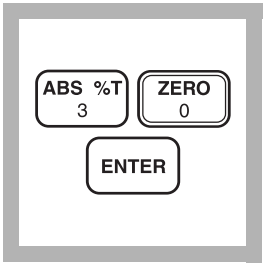
Press: **PRGM**

The display will show:

PRGM ?

Note: *Adjust the pH of stored samples before analysis.*

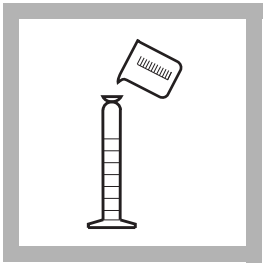
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **30 ENTER**

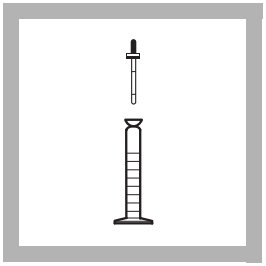
The display will show **mg/L, CaCO3** and the **ZERO** icon**.**

Note: *For alternate forms (Mg, $MgCO_3$), press the* **CONC** *key.*

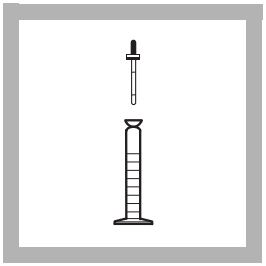


3. Pour 100 mL of sample into a 100-mL graduated mixing cylinder.

Note: *The sample temperature should be 21-29 °C (70-84 °F).*

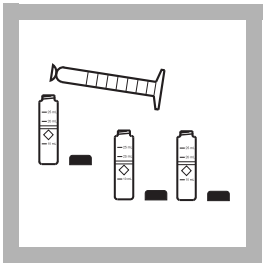


4. Add 1.0 mL of Calcium and Magnesium Indicator Solution using a 1.0-mL measuring dropper. Stopper. Invert several times to mix.



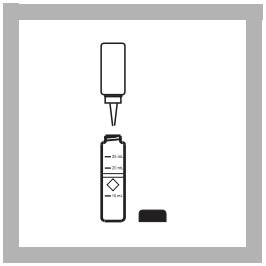
5. Add 1.0 mL of Alkali Solution for Calcium and Magnesium Test using a 1.0-mL measuring dropper. Stopper. Invert several times to mix.

Note: *If the sample turns read after adding Alkali Solution, dilute sample 1:1 and repeat analysis.*

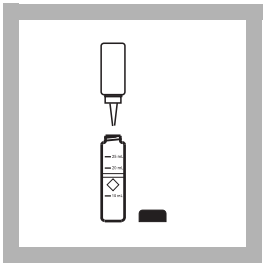


6. Pour 10 mL of the solution into each of three sample cells.

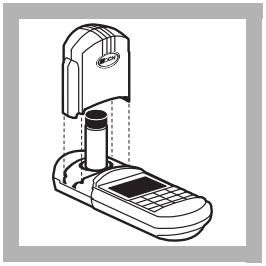
Note: *The test will detect any calcium or magnesium contamination in the mixing cylinder, measuring droppers or sample cells. To test cleanliness, repeat the test multiple times until you obtain consistent results.*



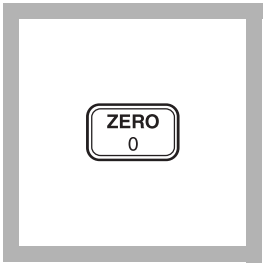
7. Add one drop of 1 M EDTA Solution to one cell (the blank). Swirl to mix.



8. Add one drop of EGTA Solution to another cell (the prepared sample). Swirl to mix.



9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

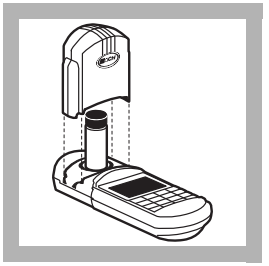


10. Press: **ZERO**

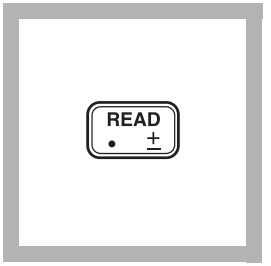
The cursor will move to the right, then the display will show:

0.00 mg/L CaCO3

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

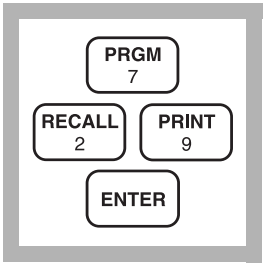


11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L magnesium hardness (as $CaCO_3$) will be displayed.



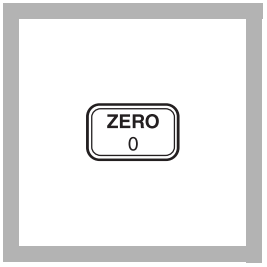
13. Without removing the cell, press:

PRGM 29 ENTER

The display will show:

PRGM ?

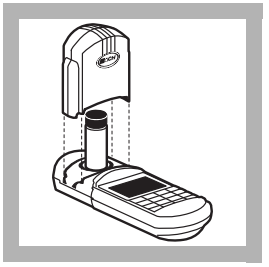
Note: *For alternate forms (Ca) press the* **CONC** *key.*



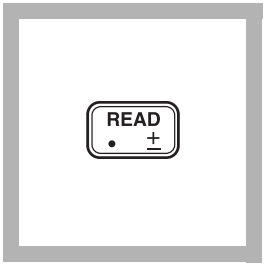
14. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L CaCO3



15. Place the third sample cell into the cell holder.



16. Press: **READ**

The cursor will move to the right, then the result in mg/L calcium hardness (as $CaCO_3$) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Note: *mg/L total hardness = mg/L Ca as $CaCO_3$ + mg/L Mg as $CaCO_3$.*

Sampling and Storage

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months. Adjust the sample pH to

between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution just before analysis. Correct the test results for volume additions; see *Correction for Volume Additions* in *Section 1* for more information.

Accuracy Check

Using a 2.00 mg/L (as $CaCO_3$) standard solution as sample, perform the hardness procedure described above. The results should be 2.00 mg/L calcium (as $CaCO_3$).

Method Performance

Precision

In a single laboratory using a standard solution of 2.00 mg/L Mg as $CaCO_3$ and 1.88 mg/L Ca as $CaCO_3$ with the instrument, a single operator obtained a standard deviation of ± 0.09 mg/L Mg as $CaCO_3$ and ± 0.08 mg/L Ca as $CaCO_3$.

Estimated Detection Limit

The estimated detection limit for program 30 is 0.13 mg/L magnesium hardness and 0.08 mg/L calcium hardness. For more information on the estimated detection limit, see *Section 1*.

Interferences

For the most accurate hardness test result, the test should be rerun on a diluted sample if the calcium is over 1.0 or the magnesium is over
0.25 mg/L as $CaCO_3$. No retesting is needed if either is below those respective concentrations.

The following cause a detectable error in test results.

Interfering Substance	Level at Which Substance Interferes
Cr^{3+}	0.25 mg/L
Cu^{2+}	0.75 mg/L
EDTA, chelated	0.2 mg/L as $CaCO_3$
Fe^{2+}	1.4 mg/L
Fe^{3+}	2.0 mg/L
Mn^{2+}	0.20 mg/L
Zn^{2+}	0.050 mg/L

Traces of EDTA or EGTA remaining in sample cells from previous tests will give erroneous results. Rinse cells thoroughly before use.

Summary of Method

The colorimetric method for measuring hardness supplements the conventional titrimetric method because it can measure very low levels of calcium and magnesium. Also some interfering metals (those listed above) in the titrimetric method are inconsequential in the colorimetric method when diluting the sample to bring it within the range of this test.

The indicator dye, calmagite, forms a purplish-blue color in a strongly alkaline solution and changes to red when it reacts with free calcium or magnesium. Calcium is chelated with EGTA to destroy any red color due to calcium and then the sample is chelated with EDTA to destroy the red color due to both calcium and magnesium. Measuring the red color in the different stages of chelation gives results as the calcium and magnesium hardness concentrations.

REQUIRED REAGENTS

	Cat. No.
Hardness Reagent Set (100 Tests)	23199-00
Includes: (1) 22417-32, (1) 22418-32, (1) 22419-26, (1) 22297-26	

Description	Quantity Required Per Test	Unit	Cat. No.
Alkali Solution for Calcium and Magnesium Test	1 mL	100 mL MDB	22417-32
Calcium and Magnesium Indicator Solution	1 mL	100 mL MDB	22418-32
EDTA Solution, 1 M	1 drop	50 mL	22419-26
EGTA Solution	1 drop	50 mL	22297-26

REQUIRED APPARATUS

Cylinder, 100-mL mixing	1	each	1896-42
Dropper, measuring, 0.5 and 1.0 mL	2	20/pkg	21247-20
Sample Cell, 10-20-25 mL, w/cap	3	6/pkg	24019-06

OPTIONAL REAGENTS

Calcium Standard Solution, 2.0 mg/L as $CaCO_3$		946 mL	20581-16
Nitric Acid, ACS		500 mL	152-49
Nitric Acid Solution, 1:1		500 mL	2540-49
Sodium Hydroxide Standard Solution 5.0 N		100 mL MDB	2450-32

OPTIONAL APPARATUS

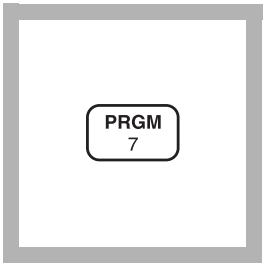
pH Meter, *sens**ion***™*1*, portable, with electrode		each	51700-10
Thermometer, –20 to 110 °C		each	26357-02

HYDRAZINE (0 to 500 µg/L)

For boiler water/feedwater, water and seawater

p-Dimethylaminobenzaldehyde Method*

Using Reagent Solution



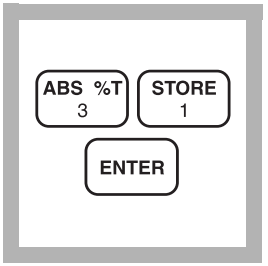
1. Enter the stored program number for hydrazine (N_2H_4).

Press: **PRGM**

The display will show:

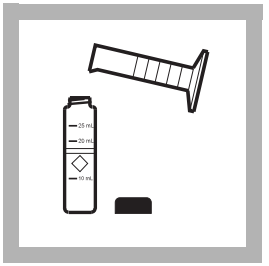
PRGM ?

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*

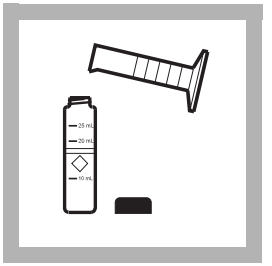


2. Press: **31 ENTER**

The display will show **µg/L, N2H4** and the **ZERO** icon**.**

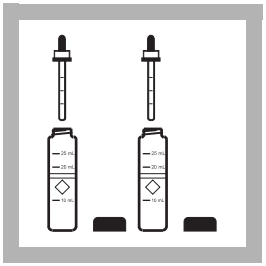


3. Pour 10.0 mL of deionized water into a sample cell (the blank) using a graduated cylinder.

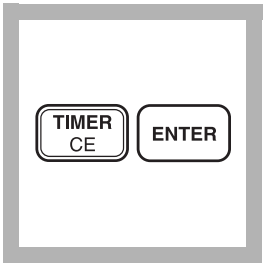


4. Pour 10.0 mL of sample into a second sample cell (the sample) using a graduated cylinder.

Note: *The sample temperature should be 21 ± 4 °C (70 ± 7 °F).*



5. Add 0.5 mL of HydraVer 2 Hydrazine Reagent to each sample cell. Cap. Invert to mix.



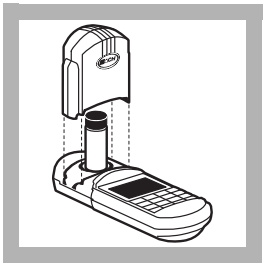
6. Press:

TIMER ENTER

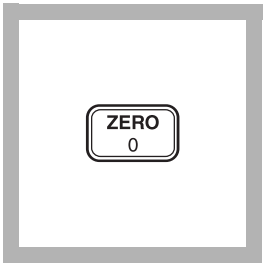
A 12-minute reaction period will begin.

Note: *Complete Steps 7-9 within 3 minutes.*

Note: *A yellow color will form if hydrazine is present. The blank will be a faint yellow color due to the HydraVer 2 reagent.*



7. Immediately after the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

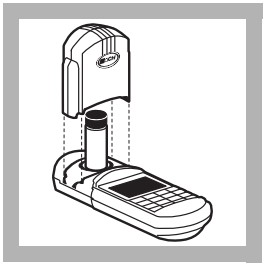


8. Press: **ZERO**

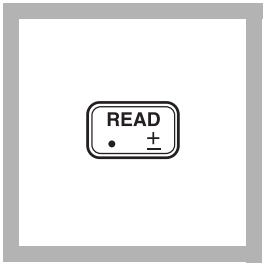
The cursor will move to the right, then the display will show:

0 µg/L N2H4

* Adapted from ASTM Manual of Industrial Water, D1385-78, 376 (1979)



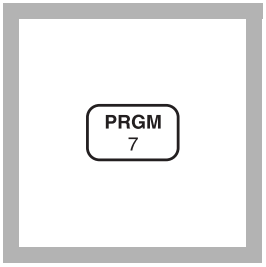
9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in µg/L hydrazine will be displayed.

Using AccuVac Ampuls



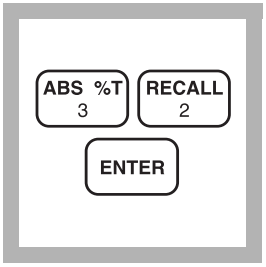
1. Enter the stored program number for hydrazine (N_2H_4)-AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

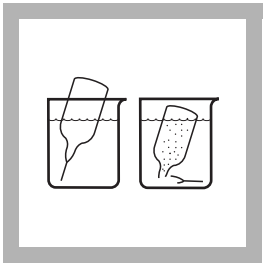
Note: *Samples must be analyzed immediately and cannot be preserved for later analysis.*



2. Press: **32 ENTER**

The display will show **µg/L, N2H4** and the **ZERO** icon.

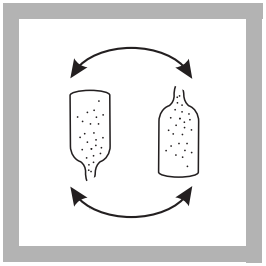
3. Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second 50-mL beaker.



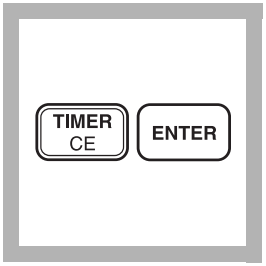
4. Fill a Hydrazine AccuVac Ampul with sample. Fill a second Hydrazine AccuVac Ampul with deionized water (the blank).

Note: *Keep the tip immersed while the ampul fills completely.*

Note: *The sample temperature should be 21 ± 4 °C (70 ± 7 °F).*



5. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.



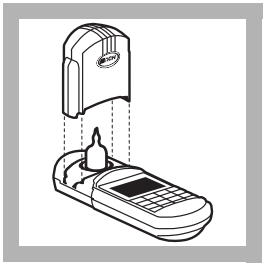
6. Press:

TIMER ENTER

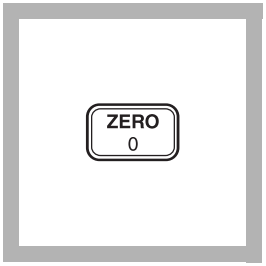
A 12-minute reaction period will begin.

Note: *Complete Steps 7-9 during this period.*

Note: *A yellow color will develop if hydrazine is present. The blank will be a faint yellow color due to the reagent.*



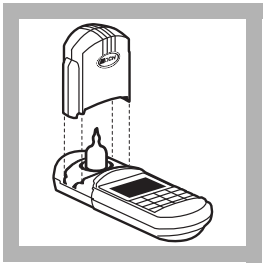
7. Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



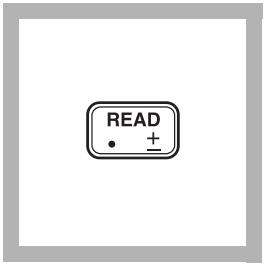
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 µg/L N2H4



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Immediately after the timer beeps, press **READ.**

The cursor will move to the right, then the result in µg/L hydrazine will be displayed.

Sampling and Storage

Collect samples in glass or plastic containers. Fill the containers completely and cap them tightly. Avoid excessive agitation or exposure to air. Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

Accuracy Check

Standard Solution Method

To assure the accuracy of the test, prepare the following solutions:

a) Prepare a 25 mg/L hydrazine stock solution by dissolving 0.1016 g of hydrazine sulfate in 1000 mL of oxygen-free deionized water. Use Class A glassware. Prepare this stock solution daily.

b) Prepare a 100 µg/L hydrazine working solution by diluting 4.00 mL of the 25 mg/L stock solution to 1000 mL with deionized oxygen-free water. Prepare just before analysis.

c) Use the working solution in place of the sample in Step 4. The result should be 100 µg/L hydrazine.

Method Performance

Precision

In a single laboratory using a standard solution of 250 µg/L hydrazine (N_2H_4) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±9 µg/L hydrazine.

In a single laboratory using a standard solution of 250 µg/L hydrazine (N_2H_4) and two lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±3 µg/L hydrazine.

Estimated Detection Limit

The estimated detection limit for program 31 is 16 µg/L N_2H_4, and the estimated detection limit for program 32 is 10 µg/L N_2H_4. For more information on the estimated detection limit, see *Section 1*.

Interferences

For highly colored or turbid samples, prepare a blank by oxidizing the hydrazine in a portion of the sample. This can be accomplished with a
1:1 mixture of deionized water and household bleach. Add two drops of this mixture to 40 mL of sample contained in a graduated mixing cylinder and invert to mix. Use this solution in Step 3, in place of deionized water, to prepare the blank.

Ammonia has no effects up to 10 mg/L ammonia. At 20 mg/L, a

positive interference occurs.

Morpholine does not interfere up to 10 mg/L.

Summary of Method

Hydrazine reacts with the p-dimethylaminobenzaldehyde from the HydraVer 2 Reagent to form a yellow color which is proportional to the hydrazine concentration.

REQUIRED REAGENTS (Using Reagent Solution)

Description	Quantity Required Per Test	Unit	Cat. No.
HydraVer 2 Hydrazine Reagent	1 mL	100 mL MDB	1790-32
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS (Using Reagent Solution)

Cylinder, graduated, 25 mL	1	each	508-40
Sample Cells, 10-, 20- and 25 mL, w/ caps	2	6/pkg	24019-06

REQUIRED REAGENTS (Using AccuVac Ampuls)

Hydrazine Reagent AccuVac Ampul	2	25/pkg	25240-25
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	2	each	500-41H

OPTIONAL REAGENTS

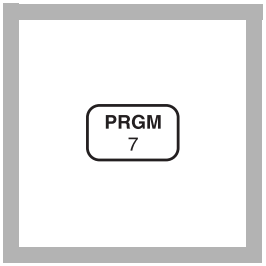
Hydrazine Sulfate, ACS		100 g	742-26

OPTIONAL APPARATUS

AccuVac Snapper Kit		each	24052-00
Balance, Analytical, 115 V, 0.1 mg		each	28014-01
Balance, Analytical, 220 V, 0.1 mg		each	28014-02
Cylinder, graduated, mixing, 25 mL		each	1896-40
Flask, volumetric, 100 mL, Class A		each	14574-42
Flask, volumetric, 1000 mL, Class A		each	14574-53
Pipet, serological, 1 mL		each	9190-02
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet, volumetric, Class A, 1.00 mL		each	14515-35
Pipet, volumetric, Class A, 4.00 mL		each	14515-04
Pipet Filler, safety bulb		each	14651-00
Thermometer, –20 to 110 °C, non-mercury		each	26357-02
Weighing Boat, 67/46 mm, 8.9 cm sq.		500/pkg	21790-00

1,10 Phenanthroline Method* (Powder Pillows or AccuVac Ampuls)

Using Powder Pillows



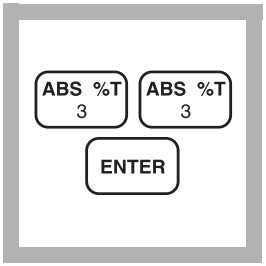
1. Enter the stored program number for Ferrous iron (Fe^{2+})-powder pillows.

Press: **PRGM**

The display will show:

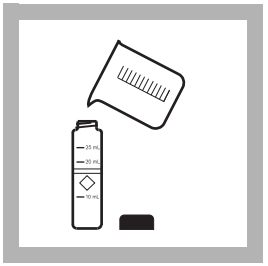
PRGM ?

Note: *Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not determined.*

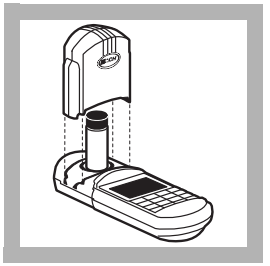


2. Press: **33 ENTER**

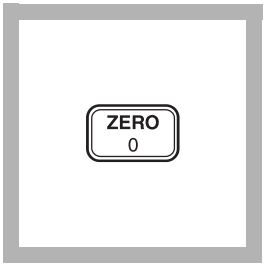
The display will show **mg/L, Fe** and the **ZERO** icon.



3. Fill a sample cell with 25 mL of sample (the blank).



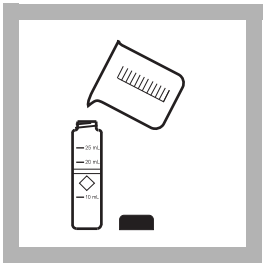
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

The cursor will move to the right, then the display will show:

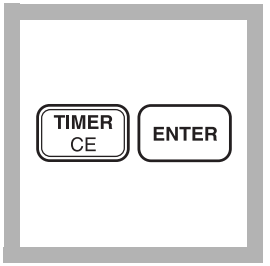
0.00 mg/L Fe



6. Fill another sample cell with 25 mL of sample.

7. Add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to mix.

Note: *Undissolved powder does not affect accuracy.*



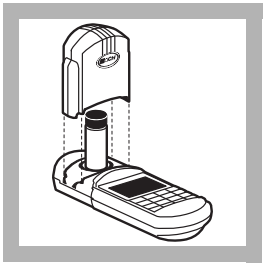
8. Press:

TIMER ENTER

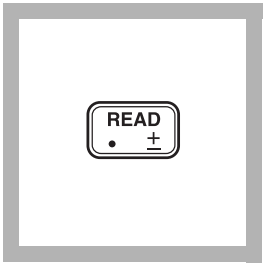
A three-minute reaction period will begin.

Note: *An orange color will form if ferrous iron is present.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

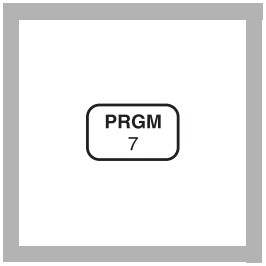


10. Press: **READ**

The cursor will move to the right, then the result in mg/L ferrous iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Using AccuVac Ampuls



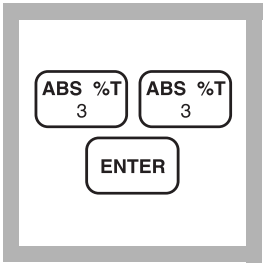
1. Enter the stored program number for ferrous iron (Fe^{2+}) AccuVac ampuls.

Press: **PRGM**

The display will show:

PRGM ?

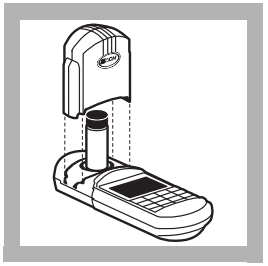
Note: *Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric, which is not determined.*



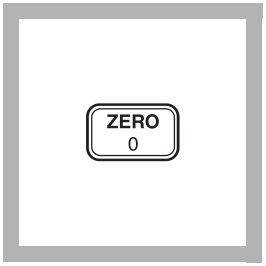
2. Press: **33 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon.

3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.



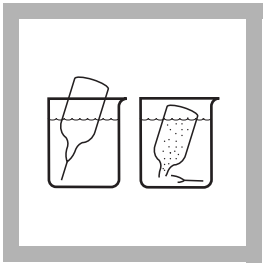
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

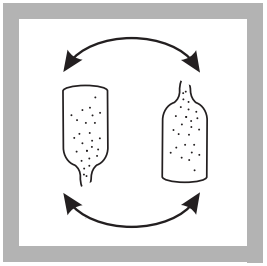
The cursor will move to the right, then the display will show:

0.00 mg/L Fe



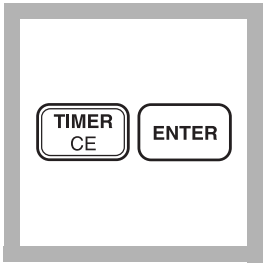
6. Fill a Ferrous Iron AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*



7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *Undissolved powder does not affect accuracy.*

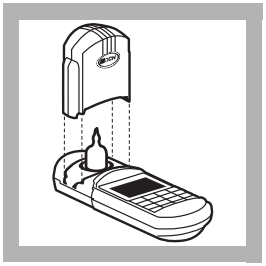


8. Press:

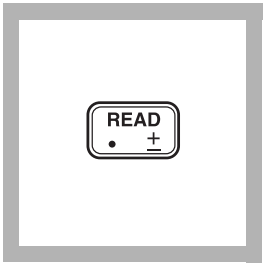
TIMER ENTER

A three-minute reaction period will begin.

Note: *An orange color will form if ferrous iron is present.*



9. Place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L ferrous iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Ferrous iron must be analyzed immediately and cannot be stored. Analyze samples as soon as possible to prevent oxidation of ferrous iron to ferric iron, which is not measured.

Accuracy Check

Standard Solution Method

Prepare a ferrous iron stock solution (100 mg/L Fe^{2+}) by dissolving 0.7022 grams of ferrous ammonium sulfate, hexahydrate, in deionized water. Dilute to 1 liter. Prepare immediately before use. Dilute 1.00 mL of this solution to 100 mL with deionized water to make a 1.00 mg/L standard solution. Prepare immediately before use.

Run the test using the 1.00 mg/L Fe^{2+} Standard Solution by following either the powder pillow or AccuVac procedure. Results should be between 0.90 mg/L and 1.10 mg/L Fe^{2+}.

Method Performance

Precision

In a single laboratory using an iron standard solution of 2.00 mg/L Fe^{2+} and two representative lots of powder pillow reagents with the instrument, a single operator obtained a standard deviation of ±0.017 mg/L Fe^{2+}.

In a single laboratory using a standard solution of 2.00 mg/L Fe^{2+} and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of ±0.009 mg/L Fe^{2+}.

Estimated Detection Limit

The estimated detection limit for program 33 (powder pillows and AccuVac Ampuls) is 0.03 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

The 1,10-phenanthroline indicator in Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The ferric iron (Fe^{3+}) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test.

REQUIRED REAGENTS & APPARATUS (USING POWDER PILLOWS)

Description	Quantity Required Per Test	Units	Cat. No.
Ferrous Iron Reagent Powder Pillows	1 pillow	100/pkg	1037-69
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (USING ACCUVAC AMPULS)

Ferrous Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25140-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Ferrous Ammonium Sulfate, hexahydrate, ACS		113 g	11256-14
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit		each	24052-00
Balance, analytical, 115 V, 0.1 mg		each	28014-01
Balance, analytical, 230 V, 0.1 mg		each	28014-02
Clippers, for opening powder pillows		each	968-00
Flask, volumetric, 100 mL, Class A		each	14574-42
Flask, volumetric, 1000 mL, Class A		each	14574-53
Pipet, volumetric, Class A, 1.00 mL		each	14515-35
Pipet Filler, safety bulb		each	14651-00
Weighing Boat, 67/46 mm, 8.9 cm square		500/pkg	21790-00

For Technical Assistance, Price and Ordering

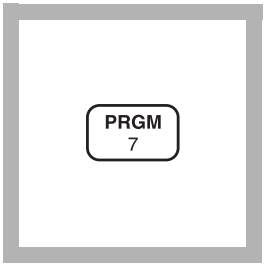
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

IRON, TOTAL (0 to 3.00 mg/L)

FerroVer Method (Powder Pillows or AccuVac Ampuls)

USEPA approved for reporting wastewater analysis (digestion is required; see Section 2*)



1. Enter the stored program number for iron (Fe) powder pillows.

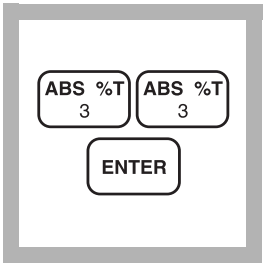
Press: **PRGM**

The display will show:

PRGM ?

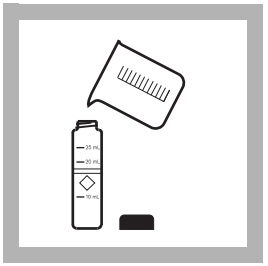
Note: *Determination of total iron requires a digestion prior to analysis (see Section 2).*

Note: *Adjust pH of stored samples before analysis.*



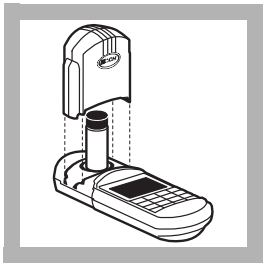
2. Press: **33 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

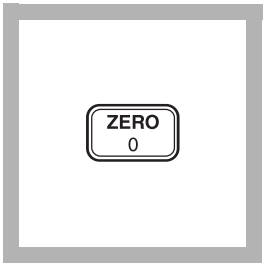


3. Fill a clean sample cell with 10 mL of sample (the blank).

Note: *For turbid samples, treat the blank with one 0.1-gram scoop of RoVer Rust Remover. Swirl to mix.*



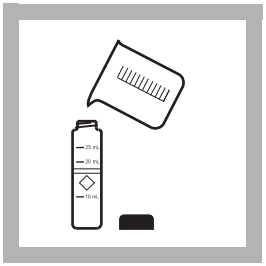
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



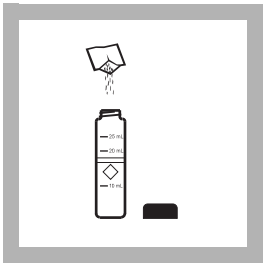
5. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L Fe

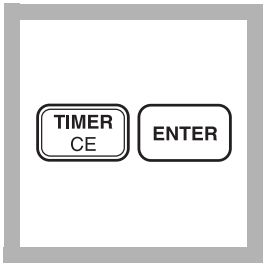


6. Fill another sample cell with 10 mL of sample.



7. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell (the prepared sample). Cap and invert to dissolve the reagent powder.

Note: *Accuracy is not affected by undissolved powder.*



8. Press:

TIMER ENTER

A three-minute reaction period will begin.

Note: *An orange color will form if iron is present.*

Note: *Samples containing visible rust should be allowed to react at least five minutes.*

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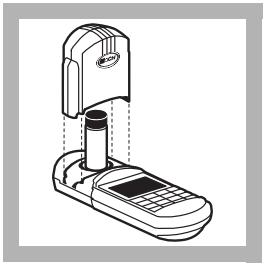
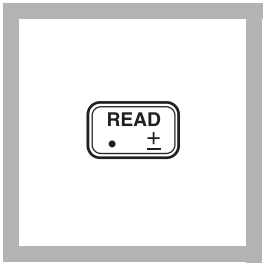
* *Federal Register*, 45 (126) 43459 (June 27, 1980). See also 40 CFR, part 136.3, Table IB.



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

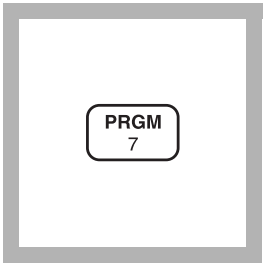


10. Press: **READ**

The cursor will move to the right, then the result in mg/L iron (Fe) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Using AccuVac Ampuls



1. Enter the stored program number for iron (Fe), AccuVac ampuls.

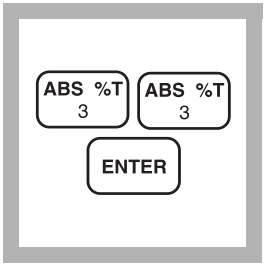
Press: **PRGM**

The display will show:

PRGM ?

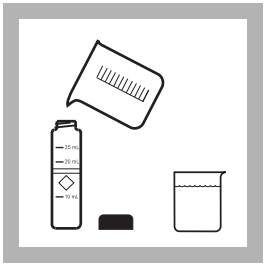
Note: *Adjust pH of stored samples before analysis.*

Note: *Determination of total iron requires a digestion prior to analysis (see Section 2).*



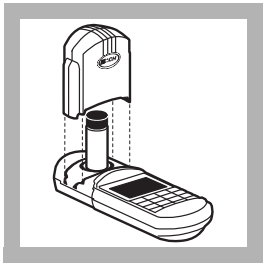
2. Press: **33 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

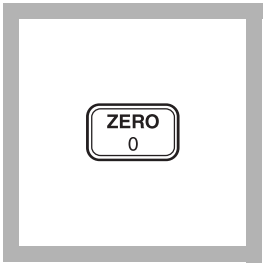


3. Fill a sample cell (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.

Note: *For turbid samples, treat the blank with one 0.1 g scoop of RoVer Rust Remover. Swirl to mix.*



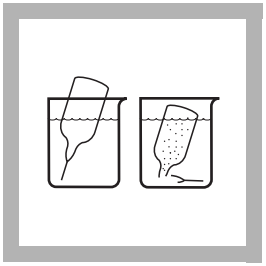
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

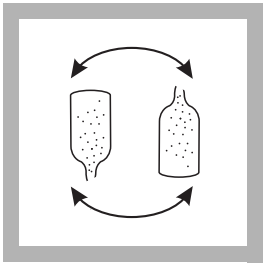
The cursor will move to the right, then the display will show:

0.00 mg/L Fe



6. Fill a FerroVer AccuVac Ampul with sample.

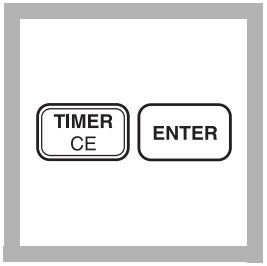
Note: *Keep the tip immersed while the ampul fills completely.*



7. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *An orange color will form if iron is present.*

Note: *Accuracy is not affected by undissolved powder.*

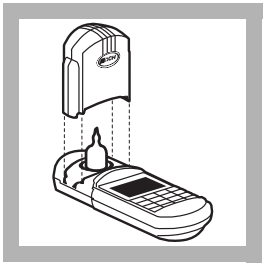


8. Press:

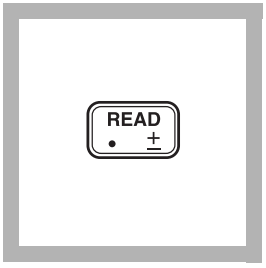
TIMER ENTER

A three-minute reaction period will begin.

Note: *Samples containing visible rust should be allowed to react at least five minutes.*



9. Place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L iron (Fe) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately. To preserve samples, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1* for more information. If only dissolved iron is to be determined, filter the sample before adding the acid.

Accuracy Check

Standard Additions Method

a) Snap the neck off a 50 mg/L Iron PourRite Ampule Standard Solution.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL samples and mix thoroughly.

c) For analysis using AccuVac Ampuls, transfer solutions to dry, clean 50-mL beakers to facilitate filling of the ampuls. For analysis with powder pillows, transfer only 10 mL of solution to the 10-mL sample cells.

d) Analyze each standard addition sample as described above. The iron concentration should increase 0.2 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

Standard Solution Method

Prepare a 1.0-mg/L iron standard by diluting 1.00 mL of Iron Standard Solution, 100 mg/L Fe, to 100 mL with deionized water. Or, dilute 1.00 mL of an Iron PourRite Ampule Standard Solution (50 mg/L) to 50 mL in a volumetric flask. Prepare this solution daily.

Run the test following the procedure for powder pillows or AccuVac Ampuls. Results should be between 0.90 mg/L and 1.10 mg/L Fe.

Method Performance

Precision

In a single laboratory, using a standard solution of 2.00 mg/L Fe and two representative lots of powder pillow reagents with the instrument, a single operator obtained a standard deviation of ±0.017 mg/L.

In a single laboratory, using a standard solution of 2.00 mg/L Fe and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of ±0.009 mg/L Fe.

Estimated Detection Limit (EDL)

The EDL for program 33 is 0.03 mg/L Fe. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatments

Interfering Substance	Interference Level and Treatment
Calcium, Ca^{2+}	No effect at less than 10,000 mg/L as $CaCO_3$
Chloride, Cl^-	No effect at less than 185,000 mg/L.
Copper, Cu^{2+}	No effect. Masking agent is contained in FerroVer Iron Reagent.
High Iron Levels	Inhibits color development. Dilute sample and retest to verify results.
Iron Oxide	Requires mild, vigorous or Digesdahl digestion (see Section 2). After digestion, adjust sample to pH 3-5 with sodium hydroxide, then analyze.
Magnesium	No effect at 100,000 mg/L as $CaCO_3$.
Molybdate, Molybdenum	No effect at 25 mg/L as Mo.
High Sulfide Levels, S^{2-}	**1.** Treat in fume hood or well-ventilated area. Add 5 mL HCl to 100 mL sample in a 250-mL Erlenmeyer flask. Boil 20 minutes. **2.** Cool. Adjust pH to 3-5 with NaOH. Re-adjust volume to 100 mL with deionized water. **3.** Analyze.

Interfering Substance	Interference Level and Treatment
Turbidity	**1.** Add 0.1 g scoop of RoVer Rust Remover to the blank in Step 3. Swirl to mix. **2.** Zero the instrument with this blank. **3.** If sample remains turbid, add three 0.2 g scoops of RoVer to a 75-mL sample. Let stand 5 minutes. **4.** Filter through a glass filter or centrifuge. **5.** Use filtered sample in Steps 3 and 6.
Sample pH (extreme)	Adjust pH to 3-5. See *Interferences* in *Section 1*.
Highly Buffered Samples	Adjust pH to 3-5. See *Interferences* in *Section 1*.

Summary of Method

FerroVer Iron Reagent reacts with all soluble iron and most insoluble forms of iron in the sample to produce soluble ferrous iron. This reacts with 1,10-phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat No.
FerroVer Iron Reagent Powder Pillows	1 pillow	100/pkg	21057-69
Sample cell, 10-20-25 mL, with screw cap	1	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

FerroVer Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25070-25
Beaker, 50 mL	1	each	500-41H

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Ammonium Hydroxide, ACS	500 mL	106-49
Drinking Water Standard, Metals, LR (Cu, Fe, Mn)	500 mL	28337-49
Drinking Water Standard, Metals, HR (Cu, Fe, Mn)	500 mL	28336-49
Hydrochloric Acid Standard Solution, 6 N	500 mL	884-49
Hydrochloric Acid, ACS	500 mL	134-49
Iron Standard Solution, 100 mg/L	100 mL	14175-42
Iron Ampule Standard, 50 mg/L	20/pkg	14254-20
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
RoVer Rust Remover	454 g	300-01
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each		24052-00
Ampule Breaker, PourRite Ampules	each		24846-00
Clippers, Shears $7^{1}/_{4}$ "	each		23694-00
Cylinder, graduated, poly, 25 mL	each		1081-40
Cylinder, graduated, poly, 100 mL	each		1081-42
Digesdahl Digestion Apparatus, 115 V	each		23130-20
Digesdahl Digestion Apparatus, 230 V	each		23130-21
Filter Discs, glass, 47 mm	100/pkg		2530-00
Filter Holder, membrane	each		2340-00
Filter Pump	each		2131-00
Flask, Erlenmeyer, 250 mL	each		505-46
Flask, filtering, 500 mL	each		546-49
Flask, volumetric, Class A, 50 mL	each		14574-41
Flask, volumetric, Class A, 100 mL	each		14574-42
Hot Plate, 4" diameter, 120 VAC	each		12067-01
Hot Plate, 4" diameter, 240 VAC	each		12067-02
pH Meter, *sens**ion***™***1***, portable, with electrode	each		51700-10
pH Indicator Paper, 1 to 11 pH	each		391-33
Pipet Filler, safety bulb	each		14651-00
Pipet, serological, 2 mL	each		532-36
Pipet, serological, 5 mL	each		532-37
Pipet, TenSette, 0.1 to 1.0 mL	each		19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg		21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg		21856-28
Pipet, volumetric, Class A, 1.00 mL	each		14515-35
Spoon, measuring, 0.1 g	each		511-00

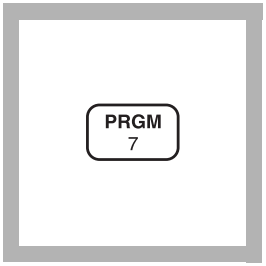
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

IRON (0 to 1.300 mg/L)

FerroZine Method*



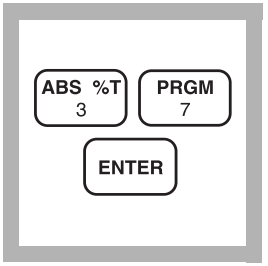
1. Enter the stored program number for iron (Fe).

Press: **PRGM**

The display will show:

PRGM ?

Note: *Adjust the pH of stored samples before analysis.*



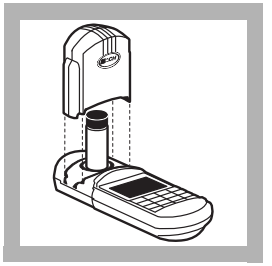
2. Press: **37 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

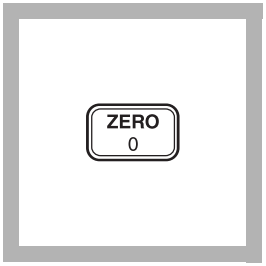
Note: *Total iron determinations need a prior digestion; use any of the three procedures given in Digestion (Section 2).*

3. Fill a sample cell with 25-mL of sample (the blank).

Note: *Rinse glassware with a 1:1 Hydrochloric Acid Solution and deionized water before use to avoid errors due to iron deposits on the glass.*



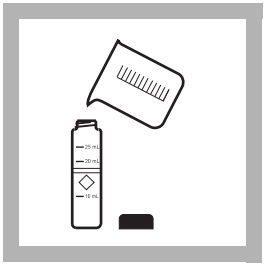
4. Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

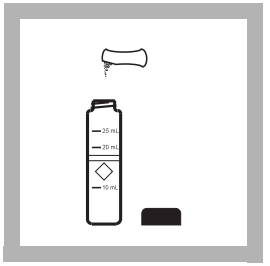
The cursor will move to the right, then the display will show:

0.000 mg/L Fe



6. Fill another sample cell with 25 mL of sample.

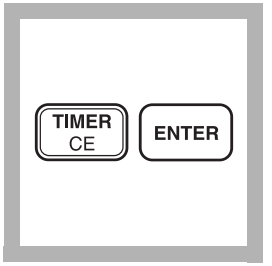
Note: *If the sample contains rust, see Interferences below.*



7. Add the contents of one FerroZine Iron Reagent Solution Pillow to the cell (the prepared sample). Cap and invert to mix.

Note: *Do not allow the clippers to come into contact with the contents of the pillow.*

Note: *If preferred , use 0.5 mL of FerroZine Iron Reagent Solution in place of the solution pillow.*



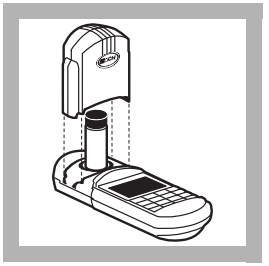
8. Press:

TIMER ENTER

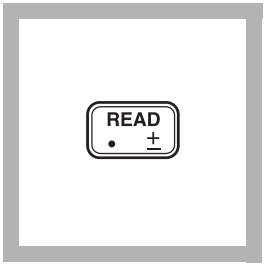
A five-minute reaction period will begin.

Note: *A violet color will develop if iron is present.*

* Adapted from Stookey, L.L., Anal. Chem., 42 (7) 779 (1970)



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with nitric acid (about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. If only dissolved iron is to be reported, filter sample immediately after collection and before the addition of nitric acid.

Before testing, adjust the sample pH to 3–5 with ammonium hydroxide, ACS. Do not exceed pH 5 as iron may precipitate. Correct test results for volume additions; see *Correction for Volume Additions* in *Section 1* for more detailed information.

Accuracy Check

Standard Additions Method

a) Snap the neck off an Iron Voluette Ampule Standard, 25 mg/L Fe.

b) Use the TenSette Pipet to add 0.1 mL of standard to the prepared sample measured in Step 10.

c) Swirl to mix and allow another five-minute reaction period, then measure the iron concentration as in Step 10.

d) Add two additional 0.1-mL standard increments, taking a

concentration reading after allowing the five-minute reaction period for each increment.

e) Each 0.1 mL of standard added should cause a 0.1 mg/L increase in the concentration reading.

f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

a) Pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a
250-mL volumetric flask.

b) Dilute to volume with deionized water. This solution should be prepared daily. Analyze the working solution according to the above procedure.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.80 mg/L iron and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.004 mg/L iron.

Estimated Detection Limit

The estimated detection limit for program 37 is 0.011 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Strong chelants, EDTA	Interfere at all levels. Use the FerroVer or TPTZ methods to test these samples. Use the TPTZ method for low iron concentrations.
Cobalt	May give slightly high results
Copper	May give slightly high results
Hydroxides	Boil the sample, with the FerroZine Iron Reagent from Step 7 added to it for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with Step 8. Return the sample volume to 25 mL with deionized water. OR Use any of the digestions in *Section 2*.
Magnetite (black iron oxide) or Ferrites	**1.** Fill a 25-mL graduated cylinder with 25 mL of sample. **2.** Transfer this sample into a 125-mL erlenmeyer flask. **3.** Add the contents of one FerroZine Iron Reagent Solution Pillow and swirl to mix. **4.** Place the flask on a hot plate or over a flame and bring to a boil. **5.** Continue boiling gently for 20 to 30 minutes. ***Note:*** *Do not allow to boil dry.* ***Note:*** *A purple color will develop if iron is present.* **6.** Return the boiled sample to the 25-mL graduated cylinder. Rinse the erlenmeyer flask with small amounts of deionized water and empty into the graduated cylinder. **7.** Return the sample volume to the 25-mL mark with deionized water. **8.** Pour this solution into a sample cell. Swirl to mix. **9.** Proceed with Step 9. OR Use any of the digestions in *Section 2*.
Rust	Boil the sample, with the FerroZine Iron Reagent from Step 7 for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with Step 8. Return the volume to 25 mL with deionized water. OR Use any of the digestions in *Section 2*.

Summary of Method

The FerroZine Iron Reagent forms a purple colored complex with trace amounts of iron in samples that are buffered to a pH of 3.5. This method is applicable for determining trace levels of iron in chemical reagents and glycols and can be used to analyze samples containing magnetite (black iron oxide) or ferrites after treatment as described in Interferences.

REQUIRED REAGENTS AND APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
FerroZine Iron Reagent Solution Pillows	1 pillow	50/pkg	2301-66
Clippers, for opening pillows	1	each	968-00
Sample Cell, 10-20-25, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Ammonium Hydroxide, ACS	500 mL	106-49	Drinking
Water Standard, Metals, LR (Cu, Fe, Mn)	500 mL		28337-49
Hydrochloric Acid Solution, 1:1 (6N)	500 mL		884-49
FerroZine Iron Reagent Solution	500 mL		2301-49
Iron Standard Solution, 100 mg/L Fe	100 mL		14175-42
Iron Standard Solution, Voluette Ampule, 25 mg/L Fe, 10 mL	16/pkg		14253-10
Nitric Acid, ACS	500 mL		152-49
Nitric Acid Solution, 1:1	500 mL		2540-49
Water, deionized	4 L		272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Clippers, shears, 7¼-inch	each	20658-00
Cylinder, graduated, 25 mL	each	508-40
Dropper, calibrated, 0.5-mL & 1.0-mL mark	6/pkg	23185-06
Flask, erlenmeyer, 125 mL	each	505-43
Flask, volumetric, 250 mL, Class A	each	14574-46
Hot plate, 3 ½" diameter, 120 V	each	12067-01
Hot plate, 3 ½" diameter, 240 V	each	12067-02
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, serological, 2 mL	each	532-36
pH Meter, *sens**ion***™***1***, portable, with electrode	each	51700-10
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96 Pipet
Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, 1.00 mL, Class A	each	14515-35
Thermometer, –20 to 110 °C, non-mercury	each	26357-02
Water Bath, with sample cell rack	each	1955-55

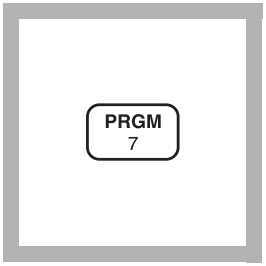
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

IRON, TOTAL (0 to 1.80 mg/L) For cooling water with molybdenum-based treatment

FerroMo™ Method*



1. Enter the stored program number for iron (Fe).

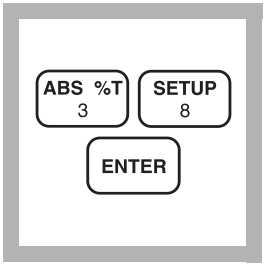
Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

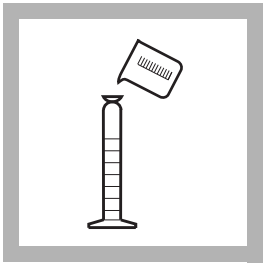
Note: *Adjust the pH of stored samples before analysis.*



2. Press: **38 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

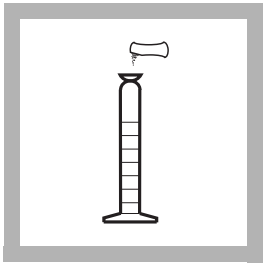
Note: *Determination of total iron requires digestion; see Section 2.*



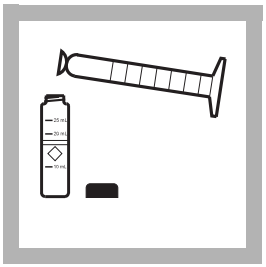
3. Fill a 50-mL graduated mixing cylinder with 50 mL of sample.

Note: *Sample pH is important in the test; see Interferences.*

Note: *Rinse glassware with 1:1 Hydrochloric Acid Solution. Rinse again with deionized water. This removes iron deposits which can cause slightly high results.*

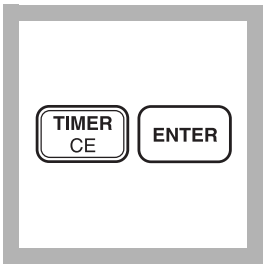


4. Add the contents of one FerroMo Iron Reagent 1 Powder Pillow to the graduated cylinder. Stopper and invert several times to mix. Remove the stopper. This is the prepared sample.



5. Transfer 25 mL of the prepared sample to a sample cell.

6. Add the contents of one FerroMo Iron Reagent 2 Powder Pillow to the sample cell. Cap the cell and shake for 30 seconds. This is the prepared sample.



7. Press:

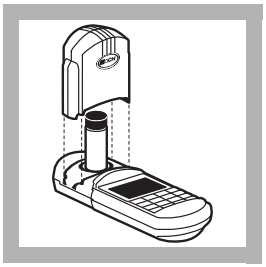
TIMER ENTER

A three-minute reaction period will begin.

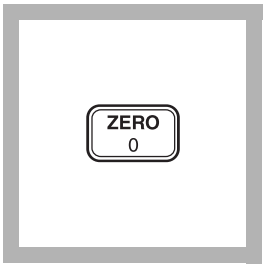
Note: *A blue color will develop if iron is present.*

8. Fill a second sample cell with 25 mL of the prepared sample from Step 4 (the blank).

* Adapted from G. Frederic Smith Chemical Company, *The Iron Reagents*, 3rd ed. (1980).



9. Insert the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

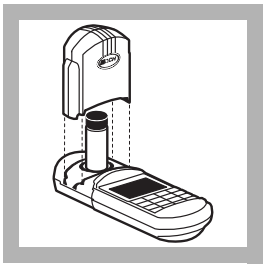


10. Press: **ZERO**

The cursor will move to the right, then the display will show:

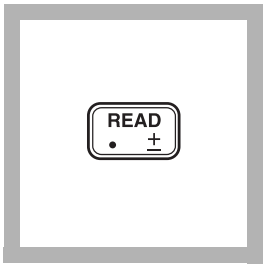
0.00 mg/L Fe

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. After the timer beeps, place the prepared sample in the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *For samples containing high levels of molybdate (≥100 mg/L), read the sample immediately after zeroing the blank.*



12. Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-cleaned plastic or glass bottles. If prompt analysis is impossible, preserve the sample by adjusting to pH 2 or less with hydrochloric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. If reporting only dissolved iron, filter the sample immediately after collection and before adding the acid.

Before analysis, adjust the sample pH to between 3 and 4 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 5 as iron may precipitate. Correct the test result for volume; see *Correction for Volume Additions* in *Section 1*.

Accuracy Check

Standard Additions Method

a) Snap the top off an Iron PourRite Ampule Standard Solution, 25 mg/L Fe.

b) Use the TenSette Pipet to add 0.2, 0.4 and 0.6 mL of standard to three 50-mL samples. Swirl gently to mix.

c) Analyze each sample as described above. The iron concentration should increase by 0.1 mg/L for each 0.2

mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more Information.

Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

a) Pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a
250-mL volumetric flask.

b) Dilute to volume with deionized water. Prepare this solution daily. Analyze this working solution according to the above procedure. Results should be between 0.36 and 0.44 mg/L Fe.

Method Performance

Precision

In a single laboratory, using a standard solution of 1.00 mg/L Fe and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of ±0.006 mg/L Fe.

Estimated Detection Limit

The estimated detection limit for program 38 is 0.03 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

Interferences

A sample pH of less than 3 or greater than 4 after reagent addition may inhibit color formation, cause the developed color to fade, or result in turbidity. Adjust the sample pH before reagent addition to between 3 and 5 using a pH meter or pH paper. Drop by drop, add an appropriate amount of acid (1.0 N Sulfuric Acid Solution) or base (1.0 N Sodium Hydroxide Standard Solution). Make volume corrections if significant amounts of acid or base are used (see *Correction for Volume Additions* in *Section 1*).

Summary of Method

FerroMo Iron Reagent 1 contains a reducing agent combined with a masking agent. The masking agent eliminates interference from high levels of molybdate. The reducing agent converts precipitated or suspended iron (rust) to the ferrous state. FerroMo Iron Reagent 2 contains the indicator combined with a buffering

agent. The indicator reacts with the ferrous iron in the sample, buffered between pH 3-4, resulting in a deep blue-purple color.

REQUIRED REAGENTS

	Cat. No.
FerroMo Reagent Set (100 tests)	25448-00
Includes: (4) 25437-68, (2) 25438-66	

Description	Quantity Required Per Test	Unit	Cat. No
FerroMo Iron Reagent 1 Powder Pillows	1 pillow	25/pkg	25437-68
FerroMo Iron Reagent 2 Powder Pillows	1 pillow	50/pkg	25438-66

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, mixing, 50 mL	1	each	1896-41
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	884-49
Hydrochloric Acid, ACS	500 mL	134-49
Iron Standard Solution, 100 mg/L Fe	100 mL	14175-42
Iron Standard Solution, PourRite Ampule, 25 mg/L Fe, 2 mL	20/pkg	24629-20
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfuric Acid Standard Solution, 1.0 N	100 mL MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	24846-00
Flask, volumetric, Class A, 250 mL	each	14574-46
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *Sens**ion**™1*, portable, with electrode	each	51700-10
Pipet Filler, safety bulb	each	14651-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 1.00 mL	each	14515-35

For Technical Assistance, Price and Ordering

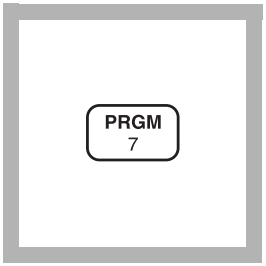
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

IRON, TOTAL (0 to 1.80 mg/L)

TPTZ Method* (Powder Pillows or AccuVac Ampuls)

Using Powder Pillows



1. Enter the stored program number for iron (Fe)- powder pillows.

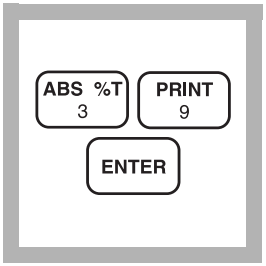
Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

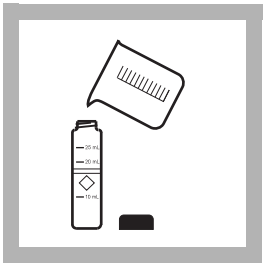
Note: *Adjust the pH of stored samples before analysis.*



2. Press: **39 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

Note: *Total iron determination needs a prior digestion. Use any of the procedures in Digestion (Section 2).*



3. Fill a sample cell with 10 mL of sample.

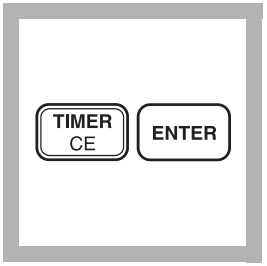
Note: *Sample pH is important in this test; see Interferences.*

Note: *Rinse glassware with a 1:1 hydrochloric acid and deionized water before use to avoid errors due to iron deposits on the glass.*

4. Add the contents of one TPTZ Iron Reagent Powder Pillow (the prepared sample). Cap and shake the cell for 30 seconds.

Note: *A blue color will develop if iron is present.*

* Adapted from G. Frederic Smith Chemical Co., *The Iron Reagents*, 3rd ed. (1980).

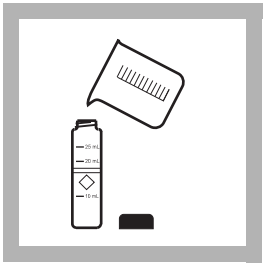


5. Press:

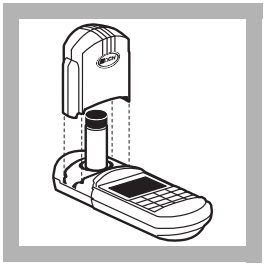
TIMER ENTER

A three-minute reaction period will begin.

Note: *Continue with Steps 6 to 8 while the timer is running.*

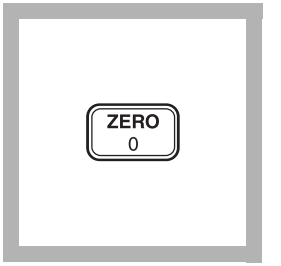


6. Fill a second sample cell with 10 mL of sample (the blank).



7. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Press* **EXIT** *to zero the instrument while the timer is running.*

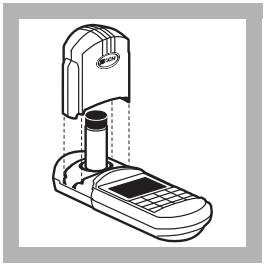


Press: **ZERO**

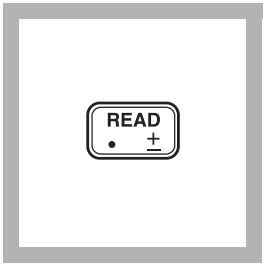
The cursor will move to the right, then the display will show:

0.00 mg/L Fe

If Reagent Blank Correction is on, the display may flash "limit". See Section 1.



8. After the timer beeps, place the prepared sample in the cell holder. Tightly cover the sample cell with the instrument cap.

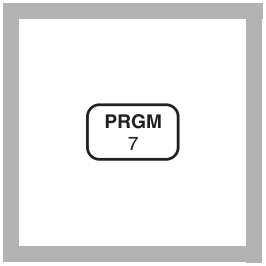


9. Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Using AccuVac Ampuls



1. Enter the stored program number for iron (Fe)- AccuVac Ampuls.

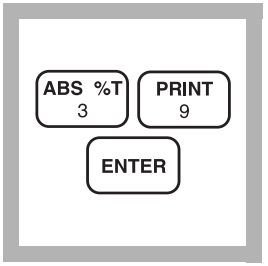
Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

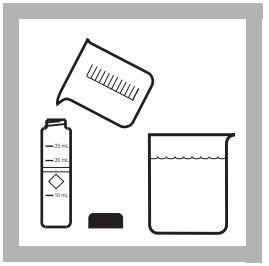
Note: *Adjust the pH of stored samples before analysis.*



2. Press: **39 ENTER**

The display will show **mg/L, Fe** and the **ZERO** icon**.**

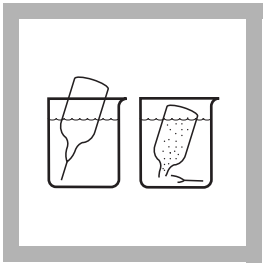
Note: *Total iron determination needs a prior digestion. Use any of the three procedures in Digestion (Section 2).*



3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

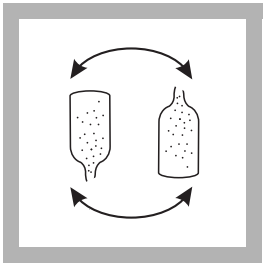
Note: *Sample pH is important in this test; see Interferences.*

Note: *Rinse glassware with a 1:1 hydrochloric acid and deionized water before use to avoid errors due to iron deposits on the glass.*



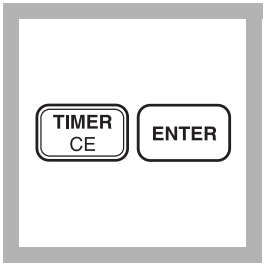
4. Fill a TPTZ Iron AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*



5. Invert the ampul (the prepared sample) repeatedly to mix. Wipe off any liquid or fingerprints.

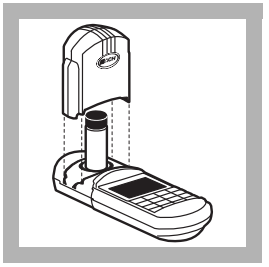
Note: *A blue color will develop if iron is present.*



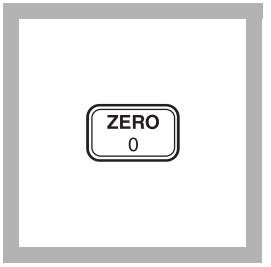
6. Press:

TIMER ENTER

A three-minute reaction period will begin.



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



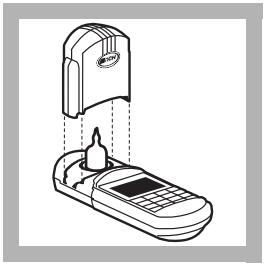
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

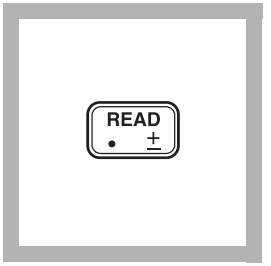
0.00 mg/L Fe

Note: *Press* **EXIT** *to zero the instrument while the timer is running.*

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. When the timer beeps, place the prepared sample into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L iron will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-washed glass or plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 2 mL per liter). Store samples preserved in this manner up to six months at room temperature. If reporting only dissolved iron, filter sample immediately after collection and before addition of nitric acid.

Before testing, adjust the pH of the stored sample to between 3 and 4 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 5 as iron may precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1*.

Accuracy Check

Standard Additions Method (Powder Pillows)

a) Snap the neck off a PourRite Iron Ampule Standard, 25 mg/L Fe.

b) Use the TenSette Pipet to add 0.1 mL of standard to the prepared sample measured in Step 10. Swirl to mix.

c) Measure the iron concentration as in Step 10. The measurement does not require the three-minute waiting period.

d) Add two additional 0.1-mL aliquots of standard, measuring the concentration after each addition. The iron concentration should increase by 0.25 mg/L for each 0.1-mL addition of standard.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method (AccuVac Ampuls)

a) Use a graduated cylinder to measure 25.0 mL of sample into each of three 50-mL beakers.

b) Snap the neck off an Iron Ampule Standard, 25 mg/L Fe.

c) Using a TenSette Pipet, add 0.1, 0.2 and 0.3 mL of standard, respectively, to the 50-mL beakers. Swirl to mix.

d) Fill a TPTZ AccuVac Ampul from each beaker.

e) Measure the concentration of each ampul according to the procedure. The iron concentration should increase by 0.1 mg/L for each 0.1 mL addition of standard.

f) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 0.4 mg/L iron working solution as follows:

a) Using Class A glassware, pipet 1.00 mL of Iron Standard Solution, 100 mg/L Fe, into a 250-mL volumetric flask.

b) Dilute to volume with deionized water. Stopper and invert repeatedly to mix. Prepare this solution daily.

Method Performance

Precision

In a single laboratory using a standard solution of 1.00 mg/L Fe and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.017 mg/L Fe.

In a single laboratory using a standard solution of 1.00 mg/L Fe and one representative lot of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.022 mg/L Fe.

Estimated Detection Limit

The estimated detection limit for program 39 is 0.04 mg/L Fe. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Cadmium	Greater than 4.0 mg/L
Chromium ($^{3+}$)	Greater than 0.25 mg/L
Chromium ($^{6+}$)	Greater than 1.2 mg/L
Cobalt	Greater than 0.05 mg/L
Color or turbidity	If the sample is turbid, add one 0.1-g scoop of RoVer Rust Remover to the blank in Step 6 (Step 3 for AccuVac procedure). Swirl to mix.
Copper	Greater than 0.6 mg/L
Cyanide	Greater than 2.8 mg/L
Manganese	Greater than 50.0 mg/L
Mercury	Greater than 0.4 mg/L
Molybdenum	Greater than 4.0 mg/L
Nickel	Greater than 1.0 mg/L
Nitrite Ion	Greater than 0.8 mg/L
pH	A sample pH of < 3 or >4 after the addition of reagent may inhibit color formation, cause the developed color to fade quickly or result in turbidity. Adjust the sample pH to 3–5 before adding reagent using a pH meter or pH paper and adding (dropwise) an appropriate amount of iron-free acid or base (i.e., 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standard Solution). Make a volume correction if significant volumes of acid or base are used.

Summary of Method

The TPTZ Iron Reagent forms a deep blue-purple color with ferrous iron. The indicator is combined with a reducing agent which converts precipitated or suspended iron, such as rust, to the ferrous state. The amount of ferric iron present can be determined as the difference between the results of a ferrous iron test and the concentration of total iron.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
TPTZ Iron Reagent Powder Pillows,	1 pillow	100/pkg	26087-99
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

REQUIRED REAGENTS (Using AccuVac Ampuls)

TPTZ Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25100-25

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41H
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

OPTIONAL REAGENTS

Drinking Water Standard, Metals, LR (Cu, Fe, Mn)	500 mL	28337-49
Drinking Water Standard, Metals, HR (Cu, Fe, Mn)	500 mL	28336-49
Hydrochloric Acid Solution, 1:1, 6.0 N	500 mL	884-49
Iron Standard Solution, 100 mg/L Fe	100 mL	14175-42
Iron Standard Solution, Ampule, 25 mg/L Fe, 2 mL	20/pkg	24629-20
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
RoVer Rust Remover	454 g	300-01
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfuric Acid Standard Solution	100 mL MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit	each	24052-00
Ampule Breaker, Ampules	each	24846-00
Cylinder, graduated, 25 mL	each	1081-40
Dropper, graduated, 0.5 and 1.0 mL marks	20/pkg	21247-20
Flask, volumetric, Class A, 250 mL	each	14574-46
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *sens**ion**™1*, portable, with electrode	each	51700-10
Pipet Filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 1.00 mL	each	14515-35

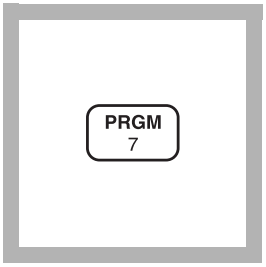
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

MANGANESE, High Range (0 to 20.0 mg/L)

Periodate Oxidation Method* USEPA approved for reporting wastewater analysis (digestion is required; see Section 2)**

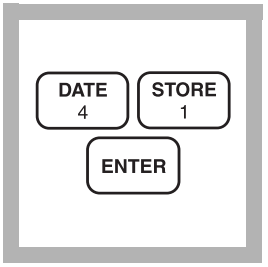


1. Enter the stored program number for manganese, periodate oxidation method.

Press: **PRGM**

The display will show:

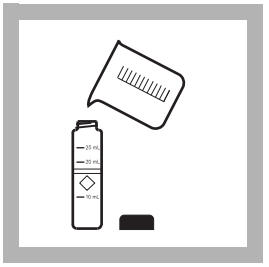
PRGM ?



2. Press: **41 ENTER**

The display will show **mg/L, Mn** and the **ZERO** icon**.**

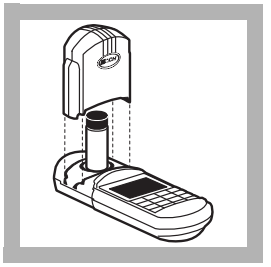
Note: *For alternate forms ($KMnO_4$, MnO_4), press the* **CONC** *key.*



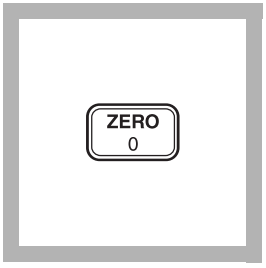
3. Fill a sample cell with 10 mL of sample (the blank).

Note: *For total manganese determination perform a digestion (see Section 2).*

Note: *Adjust the pH of stored samples before analysis.*



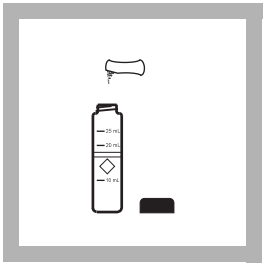
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



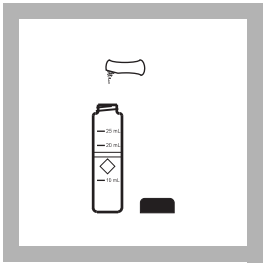
5. Press: **ZERO**

The cursor will move to the right, then the display will show:

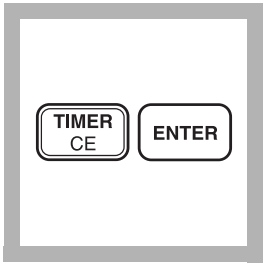
0.0 mg/L Mn



6. Remove the cell from the instrument. Add the contents of one Buffer Powder Pillow, citrate type, to the cell. Cap the cell and invert until the powder is dissolved. Remove cap.



7. Add the contents of one Sodium Periodate Powder Pillow to the sample cell (the prepared sample). Cap the sample cell. Invert for 10 seconds to mix.



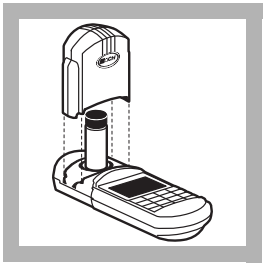
8. Press:**TIMER ENTER**

A two-minute reaction period will begin.

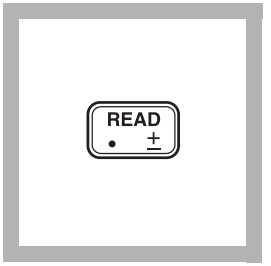
Note: *A violet color will form if manganese is present.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

** *Federal Register,* 44 (116) 34193 (June 14, 1979).



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L manganese will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in acid-washed plastic bottles. Manganese may be lost by adsorption to glass container walls. Adjust the pH to less than 2 with nitric acid (about 2 mL per liter). Preserved samples may be stored up to six months at room temperature. Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as manganese may be lost as a precipitate. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information. If only dissolved Mn is to be determined, filter before acid addition.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Manganese Voluette Ampule Standard Solution, 250 mg/L Mn.

b) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three 25-mL water samples. Swirl to mix.

c) Transfer only 10 mL of each solution to the 10-mL sample cells.

d) Analyze each standard addition sample as described in the procedure. The manganese concentration should increase 1.0 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

Standard Solution Method

Prepare a 5.0 mg/L manganese standard solution by pipetting (use a TenSette or Class A volumetric pipet) 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask. Dilute to the mark with deionized water. Or, prepare this standard by diluting 1.00 mL of a High Range Manganese Standard Voluette Ampule, 250 mg/L, to 50 mL. Prepare these solutions daily. Use these solutions as the sample in the procedure.

Method Performance

Precision

In a single laboratory, using a standard solution of 10.00 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.18 mg/L Mn.

Estimated Detection Limit

The estimated detection limit for program 41 is 0.2 mg/L Mn. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Calcium	700 mg/L
Chloride	70,000 mg/L
Iron	5 mg/L
Magnesium	100,000 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interferences* in *Section 1*.

Summary of Method

Manganese in the sample is oxidized to the purple permanganate state by sodium periodate, after buffering the sample with citrate. The purple color is directly proportional to the manganese concentration.

REQUIRED REAGENTS

	Cat. No.
High Range Manganese Reagent Set (100 tests) 10 mL	24300-00
Includes: (1) 21076-69, (1) 21077-69	

Description	Quantity Required Per Test	Unit	Cat. No.
Buffer Powder Pillows, citrate type for Manganese	1 pillow	100/pkg	21076-69
Sodium Periodate Powder Pillows for Manganese	1 pillow	100/pkg	21077-69

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Drinking Water Standard, Metals, HR (Cu, Fe, Mn)	500 mL	28336-49
Hydrochloric Acid, 6 N	500 mL	884-49
Manganese Standard Solution, 1000 mg/L Mn	100 mL	12791-42
Manganese Standard Solution, Voluette ampule, High Range, 250 mg/L Mn, 10 mL	16/pkg	14258-10
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution 1:1	500 mL	2540-49
Sodium Hydroxide Solution, 5.0 N	100 mL MDB	2450-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Flask, Erlenmeyer, 250 mL	each	505-46
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 1000 mL	each	14574-53
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable, with electrode	each	51700-10
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96Pipet
Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet Tips, for 19700-10 TenSette Pipet	50/pkg	21997-96
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00

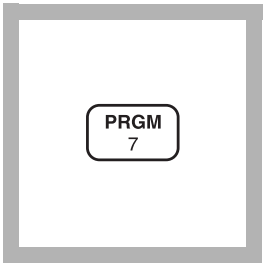
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

MANGANESE, Low Range (0 to 0.700 mg/L)

PAN Method*

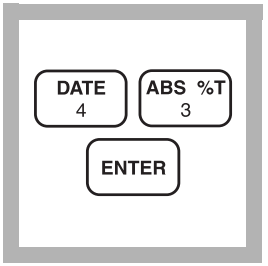


1. Enter the stored program number for low range manganese.

Press: **PRGM**

The display will show:

PRGM ?

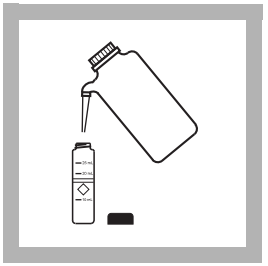


2. Press: **43 ENTER**

The display will show **mg/L, Mn** and the **ZERO** icon**.**

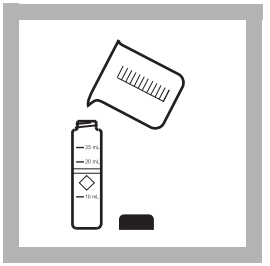
Note: *For alternate forms (MnO_4, $KMnO_4$), press the* **CONC** *key.*

Note: *Total manganese determination requires a prior digestion; see Digestion (Section 2).*

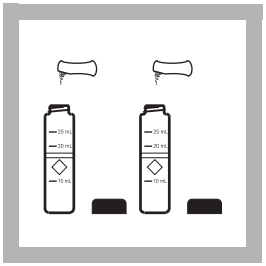


3. Fill a sample cell with 10 mL of deionized water (the blank).

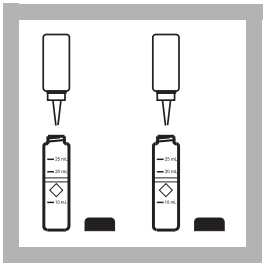
Note: *Rinse all glassware with 1:1 Nitric Acid Solution. Rinse again with deionized water.*



4. Fill another sample cell with 10 mL of sample (the prepared sample).



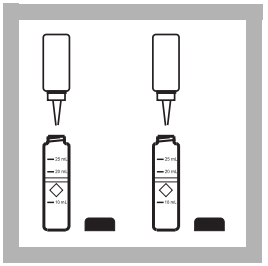
5. Add the contents of one Ascorbic Acid Powder Pillow to each cell. Swirl to mix.



6. Add 12 drops of Alkaline-Cyanide Reagent Solution to each cell. Swirl to mix.

Note: *A cloudy solution may form in some samples after reagent addition. The turbidity should dissipate after Step 8.*

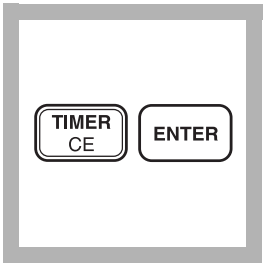
Note: *A Tensette Pipet may be used to dispense 0.4 mL of the Alkaline Cyanide Reagent.*



7. Add 12 drops of PAN Indicator Solution, 0.1%, to each sample cell. Swirl to mix.

Note: *An orange color will develop in the sample if manganese is present.*

Note: *A Tensette Pipet may be used to dispense 0.4 mL of the PAN Indicator Solution.*

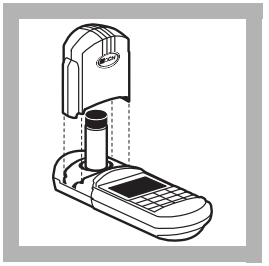


8. Press:

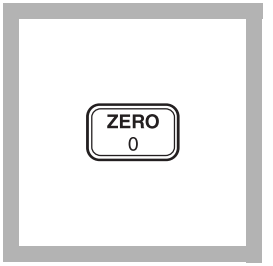
TIMER ENTER

A two-minute reaction period will begin.

* Adapted from Goto, K., et al., Talanta, 24, 752-3 (1977).



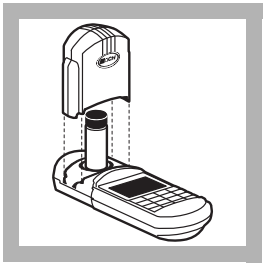
9. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



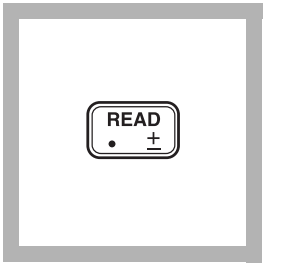
10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.000 mg/L Mn



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L manganese will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Note: *See Waste Management below for proper disposal of cyanide wastes.*

Sampling and Storage

Collect samples in a clean glass or plastic container. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the pH to 4.0-5.0 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1*.

Accuracy Check

Standard Additions Method

Note: *Volume accuracy is very important when performing standard additions with 10-mL volumes. The fill mark on the 10-mL sample cell is not intended to measure standard addition volumes.*

a) Fill three 10-mL graduated mixing cylinders with 10.0 mL of sample.

b) Snap the neck off a Manganese Voluette Ampule Standard,
10 mg/L Mn.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper and mix each thoroughly.

d) Analyze each sample as described in the procedure. The manganese concentration should increase 0.1 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Note: *An alternative to the above procedure is to pipet 10.0 mL of sample into dry sample cells before performing standard additions. A volumetric pipet or a TenSette Pipet can be used to deliver the sample volume.*

Standard Solution Method

Prepare a 0.5 mg/L manganese standard solution as follows:

a) Pipet 5.00 mL of Manganese Standard Solution, 1000 mg/L Mn, into a 1000-mL volumetric flask.

b) Dilute to the mark with deionized water. Prepare this solution daily.

c) Pipet 10.00 mL of the solution from Step b into a 100-mL volumetric flask.

d) Dilute to the mark with deionized water. This second dilution is equivalent to 0.5 mg/L Mn.

Method Performance

Precision

In a single laboratory using a standard solution of 0.5 mg/L Mn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.013 mg/L Mn.

Estimated Detection Limit

The estimated detection limit for program 43 is 0.020 mg/L Mn. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following do not interfere up to the indicated concentrations:

Substance	Suggested Treatment For Levels That Interfere
Aluminum	20 mg/L
Cadmium	10 mg/L
Cobalt	20 mg/L
Copper	50 mg/L
Hardness	300 mg/L.
Iron	If the sample contains more than 5 mg/L iron, allow 10 minutes for complete color development. Instead of performing Step 8, set the timer for 10 minutes by pressing **TIMER** twice. Then press **1000**. Press **ENTER** to start the timer.
Lead	0.5 mg/L
Magnesium	For samples containing hardness greater than 300 mg/L $CaCO_3$, add four drops of Rochelle Salt Solution to the sample after addition of the Ascorbic Acid Powder Pillow.
Nickel	40 mg/L
Zinc	15 mg/L

Waste Management

The alkaline cyanide solution contains cyanide. Cyanide solutions should be collected for disposal as reactive (D003) waste. Store all cyanide solutions in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. In case of a spill, clean up the area as outlined below:

1. Use a fume hood or self-contained breathing apparatus.
2. While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).
3. Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.
4. Flush the solution down the drain with a large excess of water.

Summary of Method

The PAN method is a highly sensitive and rapid procedure for detecting low levels of manganese. An ascorbic acid reagent is used initially to reduce all oxidized forms of manganese to Mn^{2+}. An alkaline-cyanide reagent is added to mask any potential

interferences. PAN Indicator is then added to combine with the Mn^{2+} to form an orange-colored complex.

REQUIRED REAGENTS

	Cat. No.
Manganese Reagent Set (50 tests)	26517-00
Includes: (1) 21223-26, (1) 14577-99, (1) 21224-26	

Description	Quantity Required Per Test	Unit	Cat. No.
Alkaline-Cyanide Reagent	30 drops	50 mL SCDB	21223-26
Ascorbic Acid Powder Pillows	2 pillows	100/pkg	14577-99
PAN Indicator Solution, 0.1%	42 drops	50 mL SCDB	21224-26
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508-40
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Drinking Water Standard, Metals, LR (Cu, Fe, Mn)	500 mL	28337-49
Hydrochloric Acid Solution, 1:1 (6 N)	500 mL	884-49
Manganese Standard Solution, 1000 mg/L Mn	100 mL	12791-42
Manganese Standard Sol'n, Ampule, 25 mg/L Mn, 2 mL	20/pkg.	21128-20
Nitric Acid Solution, 1:1	500 mL	2540-49
Rochelle Salt Solution.	29 mL DB	1725-33
Sodium Hydroxide Solution, 50%	500 mL	2180-49
Nitric Acid, ACS	500 mL	152-49

OPTIONAL APPARATUS

Ampule Breaker, Ampule	each	24846-00
Beaker, glass, 1000 mL	each	500-53
Cylinder, graduated, mixing, 10 mL	each	20886-38
Dropper, plastic, calibrated, 1.0 mL	20/pkg	21247-20
Flask, volumetric, Class A, 1000 mL	each	14574-53
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 10.0 mL	each	14515-38
Pipet, volumetric, Class A, 5.0 mL	each	14515-37
Pipet Filler, safety bulb	each	14651-00

For Technical Assistance, Price and Ordering

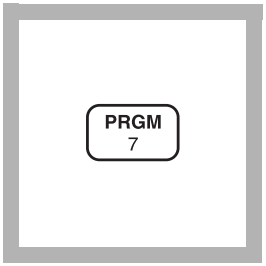
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

MOLYBDENUM, MOLYBDATE, Low Range (0 to 3.00 mg/L)

Ternary Complex Method

For boiler and cooling tower waters



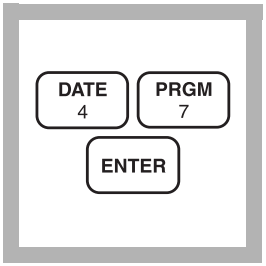
1. Enter the stored program number for molybdate molybdenum.

Press: **PRGM**

The display will show:

PRGM ?

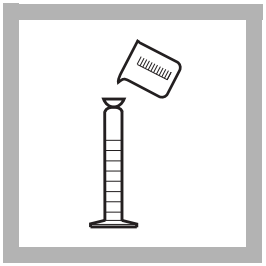
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **47 ENTER**

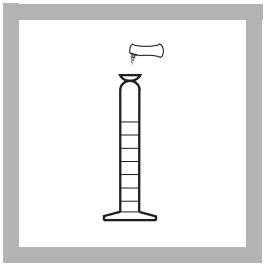
The display will show **mg/L, Mo6** and the **ZERO** icon**.**

Note: *For alternate forms (MoO_4), press the* **CONC** *key.*



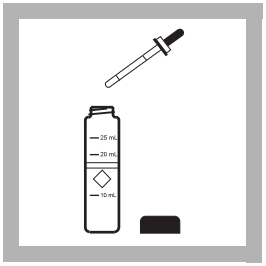
3. Fill a 25-mL mixing graduated cylinder with 20 mL of the sample.

Note: *Filter turbid samples using the labware listed under Optional Apparatus.*



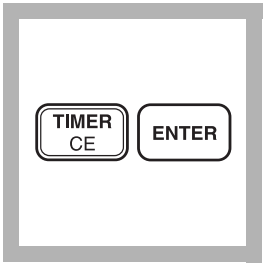
4. Add the contents of one Molybdenum 1 Reagent Powder Pillow to the graduated cylinder. Stopper. Invert the graduated cylinder several times to dissolve the reagents.

5. Pour 10 mL of the solution into a sample cell.



6. Add 0.5 mL of Molybdenum 2 Reagent to the sample cell. Swirl to mix. This is the prepared sample.

Note: *Molybdenum will cause a green color to form.*

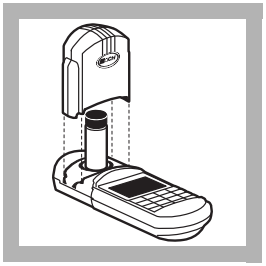


7. Press:

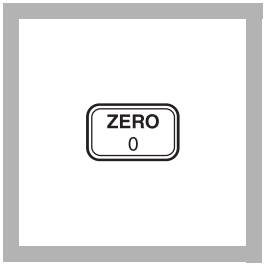
TIMER ENTER

A two-minute reaction period will begin.

8. Fill a second sample cell with 10 mL of solution from the graduated cylinder (the blank).



9. Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

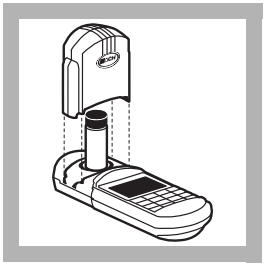


10. Press: **ZERO**

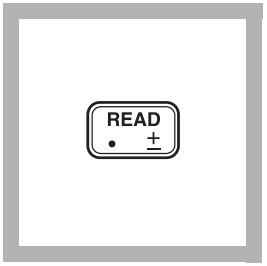
The cursor will move to the right, then the display will show:

0.00 mg/L Mo6

Note: *If Reagent Blank Correction is on, the display may flash "limit" (see Section 1).*



11. Place the developed sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L molybdate molybdenum will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in glass or plastic bottles.

Accuracy Check

Standard Addition Method

a) Add 25 mL of sample to three 25-mL mixing cylinders.

b) Snap the neck off a Molybdenum PourRite Ampule Standard Solution, 75 mg/L Mo^{6+}.

c) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to three 25-mL samples. Mix thoroughly.

d) Analyze 20 mL of each spiked sample as described in the procedure. The molybdenum concentration reading should increase by 0.3 mg/L for each 0.1 mL addition of standard.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 2.0-mg/L molybdenum standard solution by pipetting 10 mL of a 10-mg/L Molybdenum Standard Solution into a 50-

mL graduated mixing cylinder. Dilute to the mark with deionized water and mix thoroughly. Analyze 20 mL of this solution according to the procedure.

Method Performance

Precision

In a single laboratory using standard solutions of 2.00 mg/L Mo^{6+} and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.009 mg/L Mo^{6+}.

Estimated Detection Limit

The estimated detection limit for program 47 is 0.07 mg/L Mo^{6+}. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interference studies were conducted by preparing a molybdenum standard solution (2 mg/L Mo^{6+}) as well as a solution of the potential interfering ion. When the standard solution concentration changed by ±5% with a given ion concentration, the ion was considered an interference.

Table 1 Negative Interferences

Ion	Level above which it interferes (mg/L)
Iron	200
Copper	98
Chromium (Cr^{6+})	4.5[1]
Chloride	1,400
AMP (Phosphonate)	15
Phosphonohydroxyacetic Acid	32
Bisulfate	3,300
Nitrite	350
Aluminum	2
Acrylates	790
Alum	7
Lignin Sulfonate	105
Orthophosphate	4,500
Bicarbonate	5,650
EDTA	1,500
Borate	5,250
Ethylene Glycol	2% (by volume)
Sulfite	6,500
Diethanoldithiocarbamate	32

Table 1 Negative Interferences (continued)

Ion	Level above which it interferes (mg/L)
Positive Interferences	
Carbonate	1,325
Silica	600
Benzotriazole	210
Morpholine	6

[1] Read molybdenum concentration immediately after the completion of the two-minute reaction period.

Table 2 No Interference

Ion	Highest Concentration Tested (mg/L)
Zinc	400
Calcium	720
Magnesium	8,000
Manganese	1,600
Chlorine	7.5
PBTC (phosphonate)	500
Sulfate	12,800
Bisulfite	9,600
Nickel	250

Phosphonate HEDP at concentrations up to 30 mg/L will increase the apparent molybdenum concentration reading by approximately 10% (positive interference). For these samples, multiply the value obtained in step 12 by 0.9 to obtain the actual molybdenum concentration. As the concentration of HEDP increases above 30 mg/L, a decrease in the molybdenum concentration reading occurs (negative interference).

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagent and require pretreatment. Adjust the sample pH to 3-5 (use a pH meter or pH paper) by adding drops of an acid or base such as 1.0 N Sulfuric Acid Standard Solution, or 1.0 N Sodium Hydroxide Standard Solution. If a significant volume of acid or base is used, correct the result by dividing the total volume (sample + acid + base) by the original volume and multiplying the test result by this factor.

Large interferences are caused by some biocides used in cooling tower samples. Hach recommends testing the ternary complex procedure on molybdenum standards containing the specific biocides in use to determine if the ternary complex method will work with these samples.

After many samples have been analyzed, the sample cells may show a slight blue color. Rinse with Hydrochloric Acid Solution, 1:1, to eliminate the build-up.

Summary of Method

The ternary complex method for molybdenum determination is a method in which molybdate molybdenum reacts with an indicator and sensitizing agent to give a stable blue complex.

REQUIRED REAGENTS

Molybdenum Reagent Set, 20 mL sample (100 tests) 24494-00
Includes: (1) 23524-49, (1) 23525-12

Description	Quantity Required Per Test	Unit	Cat. No.
Molybdenum 1 Reagent for 20 mL sample size	1 pillow	100/pkg	23524-49
Molybdenum 2 Reagent Solution	0.5 mL	.50 mL MDB	23525-12

REQUIRED APPARATUS

Cylinder, mixing, graduated, 25 mL	1	each	1896-40
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Hydrochloric Acid Solution, 1:1, 6.0 N	500 mL	884-49
Molybdenum Standard Solution, Ampule 75 mg/L Mo^{6+}, 2 mL	20/pkg	25575-20
Molybdenum Standard Solution, 10 mg/L Mo^{6+}	100 mL	14187-42
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Cylinder, mixing, graduated, 50 mL		each	1896-41
Filter Paper, folded, 12.5 cm		100/pkg	1894-57
Funnel, poly, 65 mm		each	1083-67
pH Paper, 1-11 pH units		5 rolls/pkg	391-33
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet, volumetric, 10.00 mL, Class A		each	14515-38
Pipet Filler, safety bulb		each	14651-00
PourRite Ampule Breaker		each	24846-00

For Technical Assistance, Price and Ordering

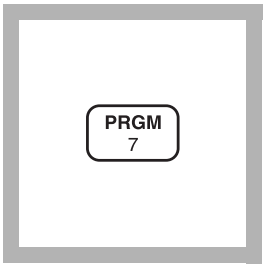
In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

MOLYBDENUM, MOLYBDATE, High Range (0 to 40.0 mg/L)

Mercaptoacetic Acid Method* — For water and wastewater

Using Powder Pillows



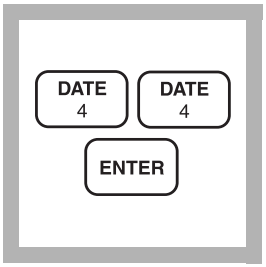
1. Enter the stored program number for high range molybdenum-powder pillows

Press: **PRGM**

The display will show:

PRGM ?

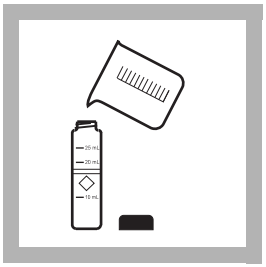
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **44 ENTER**

The display will show **mg/L, Mo6** and the **ZERO** icon**.**

Note: *For alternate form (MoO_4), press the* **CONC** *key.*

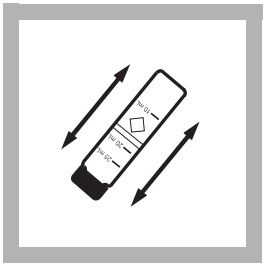


3. Fill a sample cell with 10 mL of sample.

Note: *Filter turbid samples.*

Note: *Adjust pH of stored samples before analysis.*

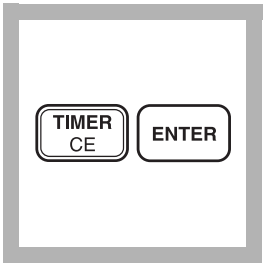
4. Add the contents of one MolyVer 1 Reagent Powder Pillow. Cap the cell and invert several times to mix.



5. Add the contents of one MolyVer 2 Reagent Powder Pillow. Cap the cell and invert several times to mix.

6. Add the contents of one MolyVer 3 Reagent Powder Pillow. Cap the cell and invert several times to mix. This is the prepared sample.

Note: *Accuracy is not affected by undissolved powder.*

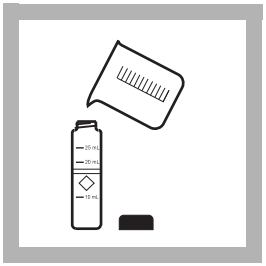


7. Press:

TIMER ENTER

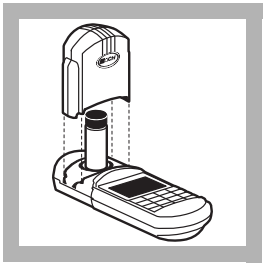
A five-minute reaction period will begin.

Note: *Molybdenum will cause a yellow color to form.*

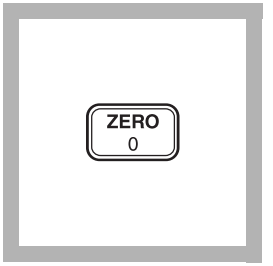


8. After the timer beeps, fill a second sample cell with 10 mL of sample (the blank).

* Adapted from Analytical Chemistry, 25(9) 1363 (1953).



9. Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

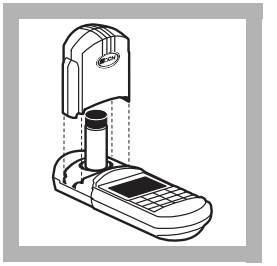


10. Press: **ZERO**

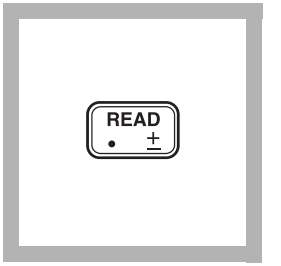
The cursor will move to the right, then the display will show:

0.0 mg/L Mo6

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



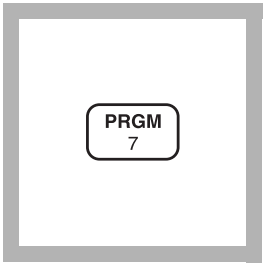
12. Press: **READ**

The cursor will move to the right, then the result in mg/L molybdenum (or alternate form) will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagents is highly recommended. See Accuracy Check.*

Using AccuVac Ampuls

Method 10046



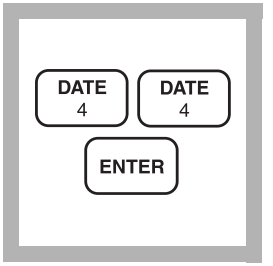
1. Enter the stored program number for high range molybdenum using AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

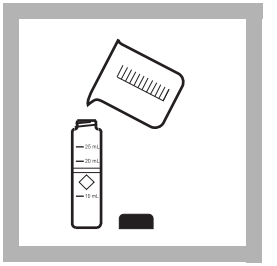
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **44 ENTER**

The display will show **mg/L, Mo6** and the **ZERO** icon**.**

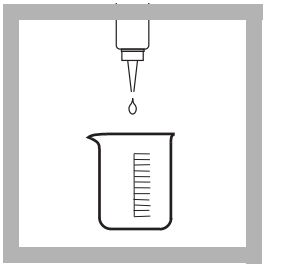
Note: *For alternate form (MoO_4), press the* **CONC** *key.*



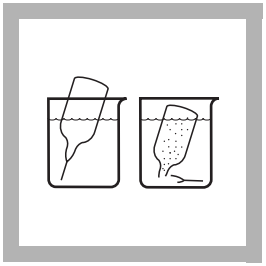
3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Filter turbid samples.*

Note: *Adjust the pH of stored samples before analysis.*

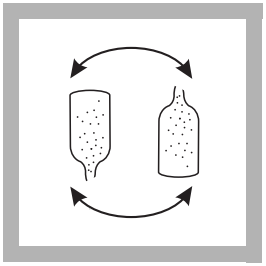


4. Add 4 drops of 0.4 M CDTA Solution to the beaker. Swirl to mix.



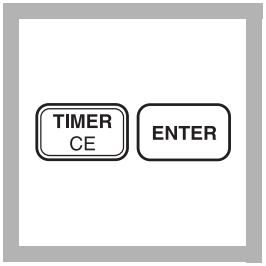
5. Fill a MolyVer 6 Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills.*



6. Invert the ampul repeatedly to mix. Wipe off any liquid or fingerprints.

Note: *Undissolved reagent will not affect the result.*

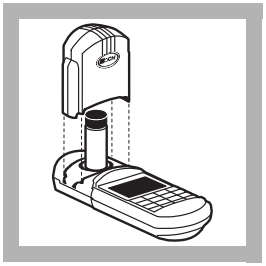


7. Press:

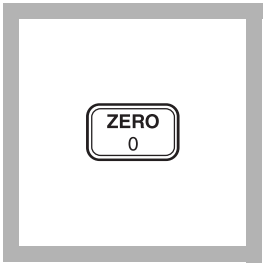
TIMER ENTER

A five-minute reaction period will begin.

Note: *If molybdenum is present a yellow color will develop.*



8. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

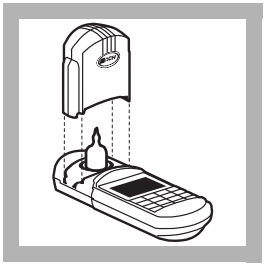


9. Press: **ZERO**

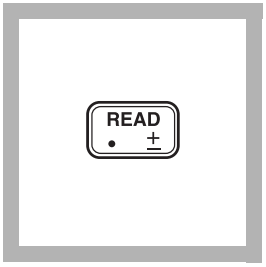
The cursor will move to the right, then the display will show:

0.0 mg/L Mo6

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the AccuVac Ampul in the cell holder. Tightly cover the ampul with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L molybdenum will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic bottles. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to 6 months at room temperature. Adjust the pH to 7 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Volume Additions* (*Section 1*) for more information.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Molybdenum Voluette Ampule Standard Solution, 500 mg/L Mo^{6+}.

c) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper each and mix thoroughly.

d) For analysis with AccuVac Ampuls, transfer solutions to dry, clean 50-mL beakers. For analysis with powder pillows, transfer only
10 mL of solution to the sample cells.

e) Analyze each standard addition sample as described in the procedure. The molybdenum concentration reading should increase 2.0 mg/L for each 0.1 mL of standard added.

f) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

Standard Solution Method

To assure the accuracy of the test, use a Molybdenum Standard Solution, 10.0 mg/L Mo^{6+}. Follow the procedure for powder pillows or AccuVac Ampuls. Results should be between 9.0 and 11.0 mg/L Mo^{6+}.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustmen*t for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 20.0 mg/L Mo^{6+} and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of ±0.3 mg/L Mo^{6+}.

In a single laboratory using a standard solution of 20.0 mg/L Mo^{6+} and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.1 mg/L Mo^{6+}.

Estimated Detection Limit

The estimated detection limit for program 44 is 0.2 mg/L Mo^{6+}. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 50 mg/L
Chromium	Greater than 1000 mg/L
Copper	Samples containing 10 mg/L copper or more will exhibit an increasing positive interference upon standing. Read these samples as soon as possible after the five-minute reaction period is complete.
Iron	Greater than 50 mg/L
Nickel	Greater than 50 mg/L
Nitrite	Interference from up to 2000 mg/L as NO_2^- can be eliminated by adding one Sulfamic Acid Powder Pillow to the sample.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see *Section 1, pH Interferences*.

Summary of Method

Powder Pillows

MolyVer 1 and 2 Reagents are added to buffer and condition the sample. MolyVer 1 contains a buffer to control the pH in addition to a chelating agent to mask interferences. MolyVer 3 provides the mercaptoacetic acid, which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

AccuVac Ampuls

The CDTA Solution masks metal interferences. The MolyVer 6 reagent provides the mercaptoacetic acid, which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

REQUIRED REAGENTS (for Powder Pillows)

	Cat. No.
Molybdenum Reagent Set, 10 mL (100 tests)	26041-00
Includes: (1) 26042-99, (1) 26043-99, (1) 26044-99	

Description	Quantity Required Per Test	Unit	Cat. No.
MolyVer 1 Reagent Powder Pillows	1 pillow	100/pkg	26042-99
MolyVer 2 Reagent Powder Pillows	1 pillow	100/pkg	26043-99
MolyVer 3 Reagent Powder Pillows	1 pillow	100/pkg	26044-99

REQUIRED REAGENTS (for AccuVac Ampuls)

MolyVer 6 Molybdenum AccuVac Reagent Set (25 tests)			25220-98
Includes: (1) 25220-25, (1) 26154-36			
CDTA Solution 0.4M	4 drops	15 mL SCDB	26154-36
MolyVer 6 Reagent AccuVac Ampuls	1 ampul	25/pkg	25220-25

REQUIRED APPARATUS (for Powder Pillows)

Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

REQUIRED APPARATUS (for AccuVac Ampuls)

Beaker, 50 mL	2	each	500-41H
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

OPTIONAL REAGENTS

Molybdenum Standard Solution, 10 mg/L Mo^{6+}	100 mL	14187-42
Molybdenum Standard Solution, Voluette Ampule, 500 mg/L Mo^{6+}, 10 mL	16/pkg	14265-10
Nitric Acid, ACS	500 mL	152-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfamic Acid Powder Pillows	100/pkg	1055-99
Water, deionized	4 L	272-56

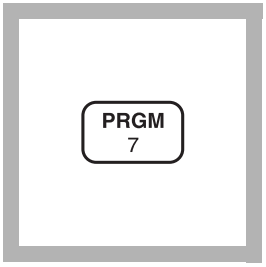
OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Cylinder, graduated, mixing, 25 mL	each	20886-40
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, Erlenmeyer, 250 mL	each	505-46
Funnel, poly, 65 mm	each	1083-67
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28

Indophenol Method* (0–4.50 mg/L Cl_2 and 0–0.50 mg/L NH_3–N)

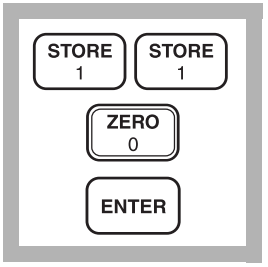
For finished chloraminated drinking water

Note: *For the most accurate chloramine results, determine a reagent blank for each new lot of reagent using deionized water instead of sample. Subtract the blank value from the final chloramine result.*



1. Enter the user program number for monochloramine.

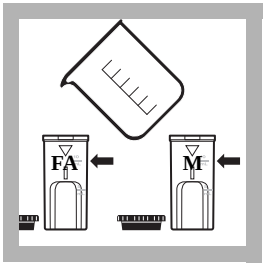
Press: **PRGM**
The display will show:
PRGM?



2. Press:
110 ENTER

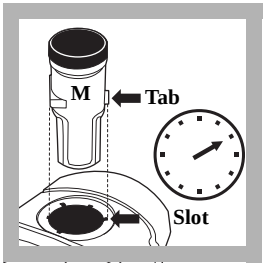
The display will show
mg/L Cl_2
and the zero icon.

Note: *For alternate forms, press the* **CONC** *key.*



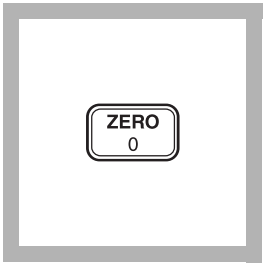
3. Fill two cells with 10 mL of sample

Label one cell "Free Ammonia" and one cell "Monochloramine".



4. Place the Monochloramine cell into the instrument so that the cell tab is at the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

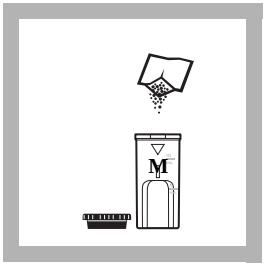
Tightly cover the sample cell with the instrument cap.



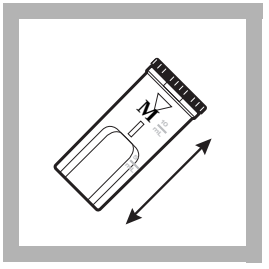
5. Press: **ZERO**

The cursor will move to the right, then the display will show:
0.00 mg/L Cl_2

Remove the cell from the instrument.

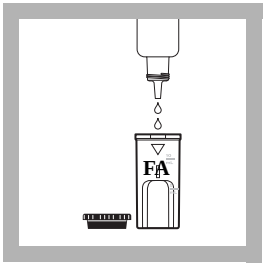


6. Add the contents of one pillow of Monochlor F to the cell for the Monochloramine measurement.



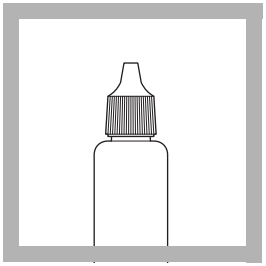
7. Cap the cell and shake for 20 seconds to dissolve the reagent.

A green color will form if monochloramine is present.

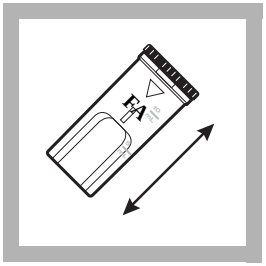


8. Add one drop of Free Ammonia Reagent Solution to the cell for Free Ammonia measurement.

* U.S. Patent 6,315,950

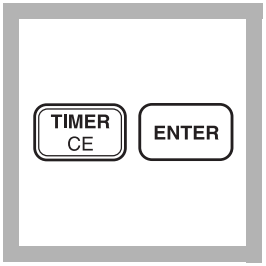


9. Cap the reagent bottle to maintain reagent performance and stability.



10. Cap the cell and mix.

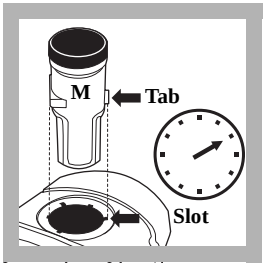
Note: *If the sample becomes cloudy by the end of the reaction period, pretreat the sample and retest. See Interferences on page 296.*



11. Press:
TIMER ENTER

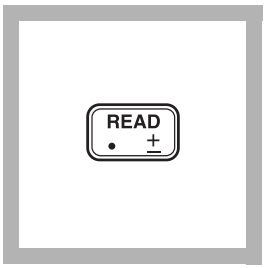
A five-minute reaction period will begin.

Note: *The color development time depends on the sample temperature. See Table 1. For accurate results allow the full reaction period to occur.*



12. When the timer expires, place the Monochloramine cell into the instrument so that the cell tab is in the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

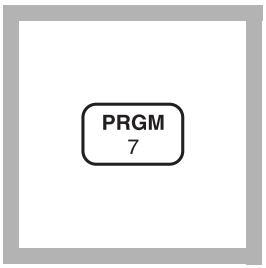
Tightly cover the sample cell with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L Monochloramine (as Cl_2 or chosen units) will be displayed.

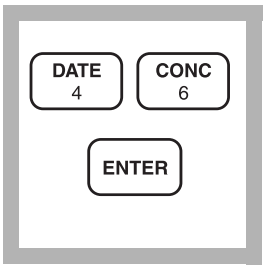
Leave the cell in the instrument.



14. Enter the stored program number for Free Ammonia.

Press: **PRGM**

The display will show PRGM?



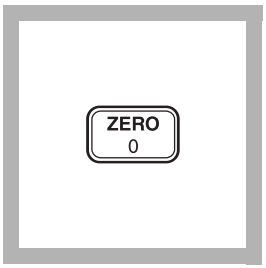
15. Press:
46 ENTER

The display will show

NH_3–N

and the zero icon.

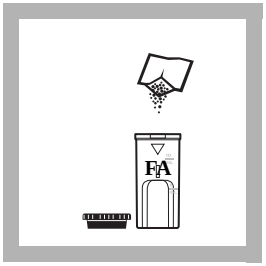
Note: *For alternate forms, press the* **CONC** *key.*



16. With the Monochloramine sample still in the cell holder, press **ZERO**.

The cursor will move to the right, then the display will show: 0.00 mg/L NH_3–N.

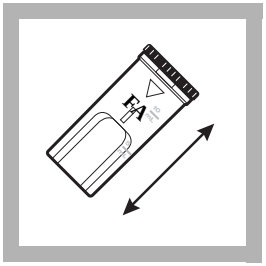
Remove the cell from the instrument.



17. Add the contents of one pillow of Monochlor F to the cell for the Free Ammonia measurement.

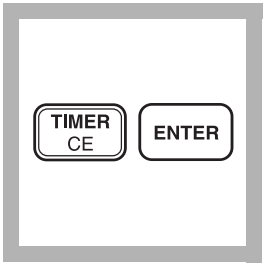
Cap and shake the cell about 20 seconds to dissolve the reagent.

Note: *The reaction period indicated in step 11 must be complete before the addition of Monochlor F to the cell for free ammonia measurement.*



18. Cap and shake the cell about 20 seconds to dissolve the reagent.

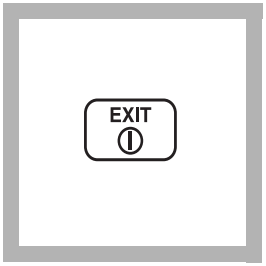
A green color will form if ammonia or monochloramine is present.



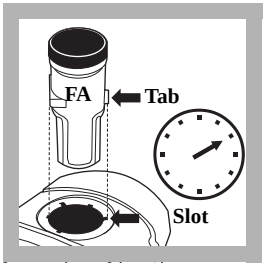
19. Press: **TIMER ENTER**

A five-minute reaction period will begin.

Note: *The color development time depends on the sample temperature. See Table 1.*

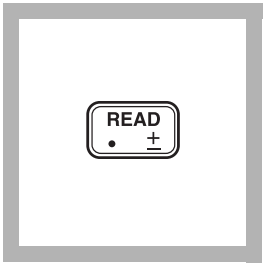


20. After the timer has expired, press: **EXIT**



21. Place the Free Ammonia cell into the instrument so that the cell tab is at the two-o'clock position. Make sure the sample cell tab is completely seated in the cell holder slot.

Tightly cover the sample cell with the instrument cap.



22. Press: **READ**

The cursor will move to the right, then the result in mg/L free ammonia as nitrogen (NH_3–N) or chosen units will be displayed.

Sampling and Storage

Collect samples in clean glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

Color Development Time

Test results are strongly influenced by sample temperature. **Both reaction periods in the procedure are the same and depend on the temperature of the sample.** The reaction periods indicated in the procedure are for a sample temperature of 18–20 °C (68–73 °F). Adjust both reaction periods according to Table 3.

Table 3 Reaction Period

Sample Temperature		Reaction Periods (Minutes)
° C	° F	
5	40	10
7	42	9
9	48	8
10	50	8
12	54	7
14	58	7
16	61	6
18	68	5
20	73	5
23	75	2.5
25	77	2
>25	>77	2

Interferences

This method is intended for finished, chloraminated drinking water samples that have a measurable combined (total) chlorine disinfectant residual. Samples where the disinfectant residual has disappeared and samples which exhibit a chlorine demand may produce low ammonia test results. Blanks and ammonia standards analyzed without a disinfectant residual must be prepared using high quality, reagent grade water.

The following do not interfere in free ammonia determination when at or below the stated concentration.

Substance	Level Tested
Aluminum	0.2 mg/L Al
Chloride	1200 mg/L Cl
Copper	1 mg/L Cu
Iron	0.3 mg/L Fe
Manganese	0.05 mg/L Mn
Nitrate	10 mg/L NO_3–N
Nitrite	1 mg/L NO_2–N
Phosphate	2 mg/L –PO_4
Silica	100 mg/L SiO_2
Sulfate	1600 ppm as $CaCO_3$
Zinc	5 ppm Zn

Samples containing high levels of both Total Hardness and Alkalinity may become turbid (cloudy) after the addition of the Free Ammonia Reagent Solution. If this occurs by the end of the first reaction period, the sample for Free Ammonia measurement must be pretreated as follows:

Note: *The sample for Monochloramine measurement does not need pretreatment.*

1. Measure 10 mL of sample into the cell for Free Ammonia measurement.
2. Add the contents of one Hardness Treatment Reagent Powder Pillow (Cat. No. 28823-46) to the sample.
3. Cap the cell and invert until the reagent is dissolved.
4. Remove the cap.

Continue with the analysis at step 2 using the pretreated sample as the Free Ammonia cell.

Accuracy Check (Monochloramine, Program 110)

1. Prepare the following monochloramine standard fresh before use.
2. Add the contents of one Buffer Powder Pillow, pH 8.3 to about 50-mL of organic-free water in a clean 100-mL Class A volumetric flask. Swirl to dissolve the powder.
3. Using a Class A volumetric pipet, transfer 2.00 mL of Nitrogen, Ammonia Standard Solution, 100 mg/L as NH_3–N into the flask.

4. Dilute to volume with organic-free water, cap and mix thoroughly. This is a 2.00 mg/L buffered ammonia standard.

5. Pipet 50.0 mL of the buffered ammonia standard into a clean 100-mL beaker. Add a stir bar.

6. Obtain a recent lot of Chlorine Solution Ampules, 50–70 mg/L, and note the actual free chlorine concentration for this lot.

7. Calculate the amount of Chlorine Solution to be added to the ammonia standard using the following equation:

$$\text{mL chlorine solution required} = \frac{455}{\text{free chlorine concentration}}$$

8. Open an ampule and, using a glass Mohr pipet, add the calculated amount of Chlorine Solution slowly to the ammonia standard, while mixing at medium speed on a stir plate.

9. Allow the monochloramine solution to mix for 1 minute after all Chlorine Solution is added.

Important Note: *Because of the strong buffer used in the preparation of this standard, it cannot be used for accuracy verification of the Free Ammonia test.*

10. Quantitatively transfer the monochloramine solution to a clean 100-mL Class A volumetric flask. Dilute to the mark with organic-free water, cap, and mix thoroughly. This is a nominal 4.5 mg/L (as Cl_2) monochloramine standard.

Use this standard within 1 hour of preparation.

Accuracy Check (Free Ammonia Test, Program 46)

Dilution water is required when testing a diluted sample and preparing standard solutions. Dilution water must be free of ammonia, chlorine and chlorine demand. A convenient source is a recirculating, deionizer system with carbon filtration which produces 18 megaohm-cm water.

Standard Additions Method

1. Measure 50 mL of sample into three 50-mL mixing cylinders.

2. Use the TenSette Pipet to add 0.3, 0.6, and 1.0 mL of Ammonium Nitrogen Standard, 10 mg/L as NH_3-N to the three samples. Mix well.

3. Analyze each spiked sample, following all steps of the Monochloramine and Free Ammonia procedure. The ammonia nitrogen concentration should increase 0.02 mg/L for each 0.1 mL of standard added.

4. If these increases do not occur, see *Standard Additions (Section 1 of the DR/890 Procedures Manual)* for more information.

Standard Solution Method

Prepare a 0.20 mg/L ammonia nitrogen standard by diluting 2.00 mL of the Ammonia Nitrogen Standard Solution, 10 mg/L, to 100 mL with dilution water. Or, using the TenSette Pipet, prepare a 0.20 mg/L ammonia nitrogen standard by diluting 0.4 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as NH_3–N, to 100 mL with dilution water. Analyze the standard solution, following all steps of the Monochloramine and Free Ammonia procedure.

Method Performance

Monochloramine Test

Precision

In a single laboratory, using a monochloramine standard solution of 2.10 mg/L Cl_2 and representative lots of reagent, a single operator obtained a standard deviation of ± 0.12 mg/L Cl_2.

Estimated Detection Limit

The estimated detection limit for Method 10171 is 0.05 mg/L Cl_2.

Free Ammonia Test

Precision

In a single laboratory using a solution containing 1.80 mg/L Cl_2 plus 0.20 mg/L ammonia nitrogen (NH_3–N) and two representative lots of reagent with the DR/890, a single operator obtained a standard deviation of ± 0.01 mg/L N for seven replicates.

Estimated Detection Limit

The estimated detection limit for program 46 is 0.02 mg/L N.

For more information on the estimated detection limit, see *Section 1* of the *DR/850 or DR/890 Procedure Manual*.

Summary of Method

Monochloramine (NH_2Cl) and "free ammonia" (NH_3 and NH_4^+) can exist in the same water sample. Added hypochlorite combines with free ammonia to form more monochloramine. In the presence of a cyanoferrate catalyst, monochloramine in the sample reacts with a substituted phenol to form an intermediate monoimine compound. The intermediate couples with excess substituted phenol to form a green-colored indophenol, which is proportional to the amount of monochloramine present in the sample. Free ammonia is determined by comparing the color intensities, with and without added hypochlorite.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet (MSDS) for information specific to the reagent used.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Free Ammonia Reagent Set (50 tests)			
Includes: (1) 28022-99, (1) 28773-36			28797-00
Free Ammonia Reagent Solution	1 drop	4 mL SCDB	28773-36
Monochlor F Reagent Pillows	2 pillows	100/pkg	28022-99

REQUIRED APPARATUS

Sample Cell, 1-cm/10-mL, with cap	2	2/pkg	48643-02

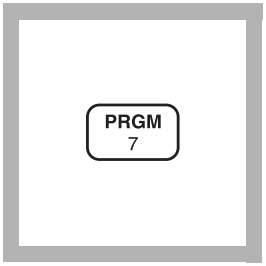
OPTIONAL REAGENTS

Buffer, pH 8.3, Powder Pillows	25/pkg	898-68
Chlorine Solution, Voluette® Ampule	16/pkg	14268-10
Hardness Treatment Reagent Pillows (1 per test)	50/pkg	28823-46
Nitrogen Ammonia Standard Solution, 10 mg/L as NH_3–N	500 mL	153-49
Nitrogen Ammonia Standard Ampule, 50 mg/L as NH_3–N, 10 mL	16/pkg	14791-10
Nitrogen Ammonia Standard Solution, 100 mg/L as NH_3–N	500 mL	24065-10

OPTIONAL APPARATUS

Description	Per Test	Unit	Cat. No.
Ampule Breaker Kit		each	21968-00
Beaker, 100 mL, Polypropylene		each	1080-42
Beaker, 100 mL, Glass		each	500-42H
Cylinder, 50 mL, mixing		each	20886-41
Flask, Volumetric, Class A, 100 mL		each	14574-42
Pipet Filler, Safety Bulb		each	14651-00
Pipet, TenSette®, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet, Mohr, Glass, 10 mL		each	20934-38
Pipet, Volumetric, Class A, 2.0 mL		each	14515-36
Pipet, Volumetric, Class A, 50.00 mL		each	14515-41
Scissors		each	28831-00
Stir Bar, Octagonal		each	20953-53
Stirrer, Magnetic		each	23436-00
Thermometer, –10 to 110 °C		each	1877-01
Wipers, Disposable Kimwipes®, 30 x 30 cm, 280/box		box	20970-01

PAN Method*

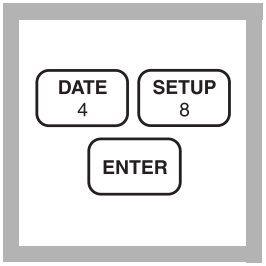


1. Enter the stored program number for nickel (Ni), PAN method.

Press: **PRGM**

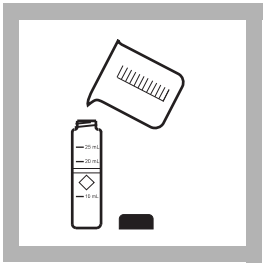
The display will show:

PRGM ?



2. Press: **48 ENTER**

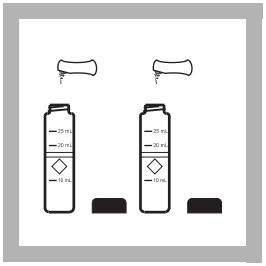
The display will show **mg/L, Ni** and the **ZERO** icon**.**



3. Fill a sample cell with 25 mL of sample (the prepared sample).

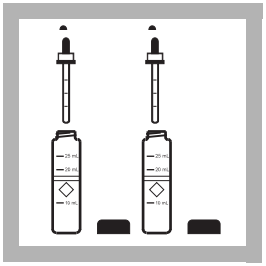
Note: *If sample is less than 10 °C (50 °F), warm to room temperature before analysis. Adjust the pH of stored samples.*

4. Fill a second sample cell with 25 mL of deionized water (the blank).



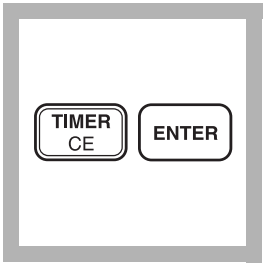
5. Add the contents of one Phthalate-Phosphate Reagent Powder Pillow to each cell. Cap. Invert several times to mix.

Note: *If sample contains iron (Fe^{3+}), all the powder must be dissolved completely before continuing with Step 6.*



6. Add 1.0 mL of 0.3% PAN Indicator Solution to each cell. Cap. Invert several times to mix.

Note: *Use the plastic dropper provided.*

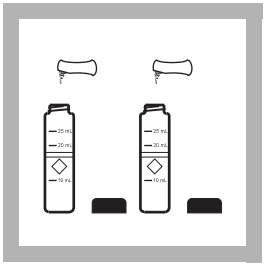


7. Press:

TIMER ENTER

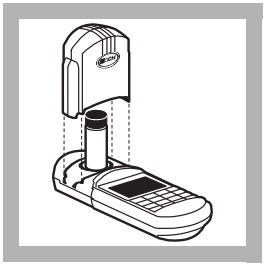
A 15-minute reaction period will begin.

Note: *The sample solution color may vary from yellowish-orange to dark red. The blank should be yellow.*

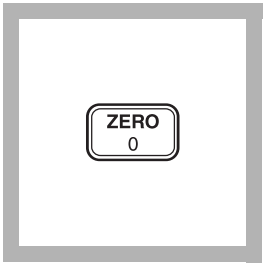


8. After the timer beeps, add the contents of one EDTA Reagent Powder Pillow to each cell. Cap. Invert several times to dissolve the reagent.

* Adapted from Watanabe, H., Talanta, 21 295 (1974)



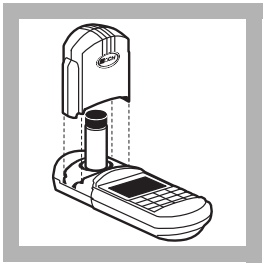
9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



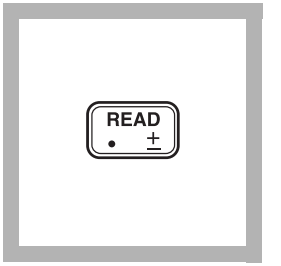
10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.000 mg/L Ni



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L nickel will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in acid-washed plastic bottles. Adjust the sample pH to 2 or less with nitric acid (about 5 mL per liter). Preserved samples can be stored up to six months at room temperature. Adjust the sample pH to between 3 and 8 with 5.0 N Sodium Hydroxide Standard Solution just before analysis. Do not exceed pH 8 as this may cause some loss of nickel as a precipitate. Correct test results for volume additions, see *Correcting for Volume Additions*, (*Section 1*) for more information.

Accuracy Check

Standard Solution Method

Prepare a 0.5 mg/L nickel standard solution by diluting 10.0 mL of a 5 mg/L working stock solution to 100 mL in a 100-mL volumetric flask. The working stock solution should be prepared daily by diluting 5.00 mL of Nickel Standard Solution, 1000 mg/L as Ni, to 1000 mL with deionized water.

Or, using the TenSette Pipet, add 0.2 mL of a Nickel Voluette Ampule Standard Solution, 300 mg/L Ni, into a 100-mL volumetric flask. Dilute to volume with deionized water. This is a 0.6 mg/L standard solution.

Method Performance

Precision

In a single laboratory using a standard solution of 0.50 mg/L nickel and two representative lots of reagent with the instrument,

a single operator obtained a standard deviation of ±0.008 mg/L nickel.

Estimated Detection Limit

The estimated detection limit for program 48 is 0.013 mg/L Ni. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Interfering Substance	Interference Level
Al^{3+}	32 mg/L
Ca^{2+}	1000 mg/L as ($CaCO_3$)
Cd^{2+}	20 mg/L
Cl^-	8000 mg/L
Co	Causes a positive interference at all levels.
Cr^{3+}	20 mg/L
Cr^{6+}	40 mg/L
Cu^{2+}	15 mg/L
F^-	20 mg/L
Fe^{3+}	10 mg/L
Fe^{2+}	Interferes directly and must not be present.
K^+	500 mg/L
Mg^{2+}	400 mg/L
Mn^{2+}	25 mg/L
Mo^{6+}	60 mg/L
Na^+	5000 mg/L
Pb^{2+}	20 mg/L
Zn^{2+}	30 mg/L

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and required sample pretreatment; see *pH Interferences (Section 1)*.

Chelating agents, such as EDTA, interfere. Use either the Digesdahl or vigorous digestion (*Section 2*) to eliminate this interference.

Summary of Method

After buffering the sample and masking any Fe^{3+} with pyrophosphate, the nickel is reacted with 1-(2-Pyridylazo)-2-Naphthol indicator.

The indicator forms complexes with most metals present. After color development, EDTA is added to destroy all metal-PAN complexes except nickel and cobalt.

REQUIRED REAGENTS

	Cat. No.
Nickel Reagent Set, 25 mL sample (100 tests)	22426-00
Includes: (2) 7005-99, (4) 21501-66, (2) 21502-32	

Description	Quantity Required Per Test	Unit	Cat. No.
EDTA Reagent Powder Pillows	2 pillows	100/pkg	7005-99
Phthalate-Phosphate Reagent Powder Pillows	2 pillows	50/pkg	21501-66
P.A.N. Indicator Solution, 0.3%	2 mL	100 mL MDB	21502-32
Water, deionized	10 mL	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, mixing, 25 mL	1	each	20886-40
Sample Cell, 10-20-25, w/caps	2	6/pkg	24019-06

OPTIONAL REAGENTS

Nickel Standard Solution, 1000 mg/L Ni		100 mL	14176-42
Nickel Standard Solution, Voluette Ampule, 300 mg/L Ni, 10 mL		16/pkg	14266-10
Nitric Acid, ACS		500 mL	152-49
Nitric Acid Solution, 1:1		500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N		100 mL MDB	2450-32

OPTIONAL APPARATUS

Ampule Breaker Kit		each	21968-00
Flask, volumetric, Class A, 100 mL		each	14574-42
Flask, volumetric, Class A, 1000 mL		each	14574-53
pH Paper, 1 to 11 pH units		5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable, with electrode		each	51700-10
Pipet, serological, 1 mL		each	9190-02
Pipet, serological, 5 mL		each	532-37
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
Pipet, volumetric, Class A, 5.0 mL		each	14515-37
Pipet, volumetric, Class A, 10.0 mL		each	14515-38
Pipet Filler, safety bulb		each	14651-00
Thermometer, –20 to 110 °C, non-mercury		each	26357-02

For Technical Assistance, Price and Ordering

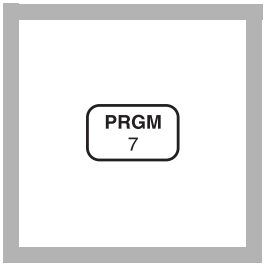
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITRATE, High Range (0 to 30.0 mg/L NO_3^--N) For water, wastewater, and seawater*

Cadmium Reduction Method (Using Powder Pillows or AccuVac Ampuls)

Using Powder Pillows



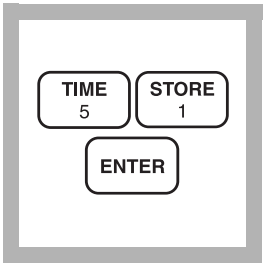
1. Enter the stored program number for high range nitrate nitrogen (NO_3^-–N) powder pillows.

Press: **PRGM**

The display will show:

PRGM ?

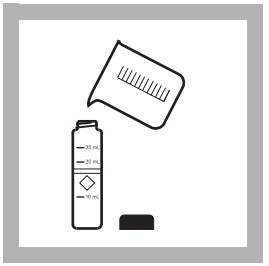
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **51 ENTER**

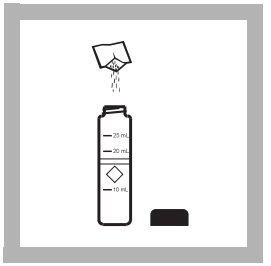
The display will show **mg/L, NO3-N** and the **ZERO** icon**.**

Note: *For alternate forms (NO_3), press the* **CONC** *key.*



3. Fill a sample cell with 10 mL of sample.

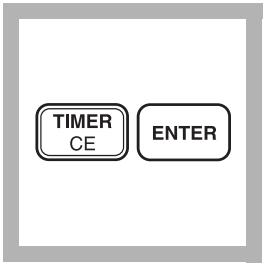
Note: *Adjust the pH of stored samples before analysis.*



4. Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow to the sample cell (the prepared sample). Cap the sample cell.

Note: *It is important to remove all of the powder from the foil pillow. Tap the pillow until no more powder pours out.*

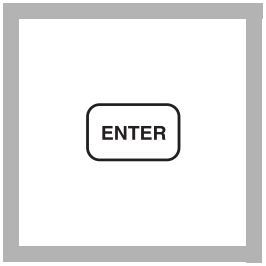
* Seawater requires a manual calibration; see Interferences.



5. Press:
TIMER ENTER

A one-minute reaction period will begin. Shake the sample cell vigorously until the timer beeps.

Note: *It is important to shake the cell vigorously. Shaking time and technique influence color development. For most accurate results, do successive tests on a standard solution and adjust the shaking time to obtain the correct result.*



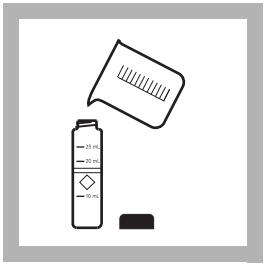
6. After the timer beeps, the display will show:
5:00 TIMER 2

Press: **ENTER**

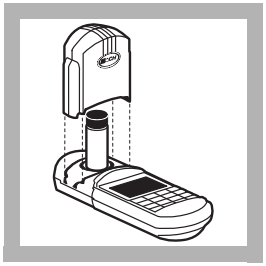
A five-minute reaction period will begin.

Note: *A deposit will remain after the reagent dissolves and will not affect test results.*

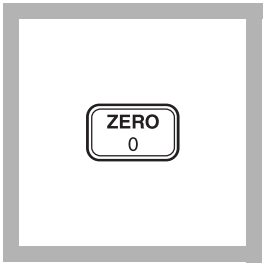
Note: *An amber color will develop if nitrate nitrogen is present.*



7. Fill another cell with 10 mL of sample (the blank). Wipe off any fingerprints or liquid.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

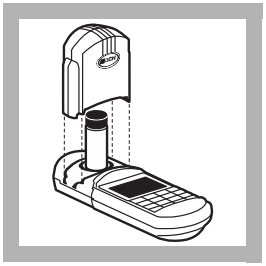


9. When the timer beeps, press **ZERO.**

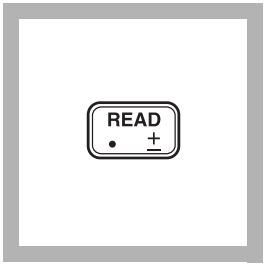
The cursor will move to the right, then the display will show:

0.0 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



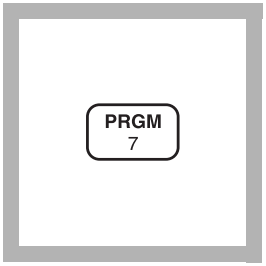
11. Press: **READ**

The cursor will move to the right, then the result in mg/L NO_3-N (or alternate form) will be displayed.

Note: *Use of the Standard Adjust feature for each new lot of reagent is highly recommended. See* Accuracy Check.

Note: *Rinse the sample cell immediately after use to remove all cadmium particles. Save the spent sample for proper hazardous waste disposal for cadmium.*

Using AccuVac Ampuls



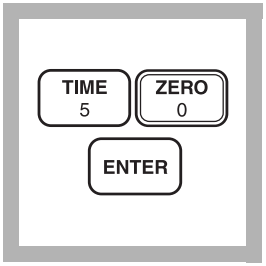
1. Enter the stored program number for high range nitrate nitrogen (NO_3^-–N) AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

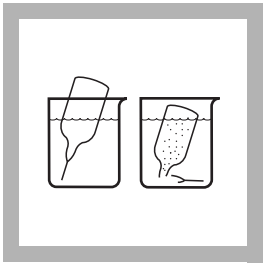
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **50 ENTER**

The display will show **mg/L, NO3-N** and the **ZERO** icon.

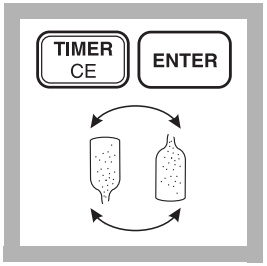
Note: *For alternate forms (NO_3), press the* **CONC** *key.*



3. Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitraVer 5 Nitrate AccuVac Ampul with sample. Place a stopper over the tip of the ampul.

Note: *Keep the tip immersed while the ampul fills. The ampul will not fill completely.*

Note: *Adjust the pH of stored samples before analysis.*

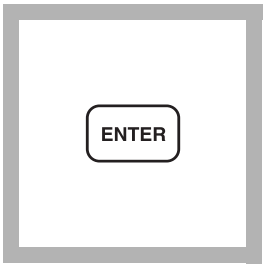


4. Press:

TIMER ENTER

A one-minute mixing period will begin. Invert the ampul repeatedly back and forth until the timer beeps. Wipe off any liquid or fingerprints.

Note: *Mixing time and technique influence color development. For most accurate results, do successive tests on a standard solution and adjust the mixing time to obtain the correct result.*



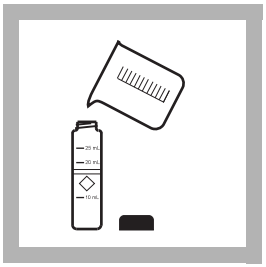
5. The display will show: **5:00 TIMER 2**

Press: **ENTER**

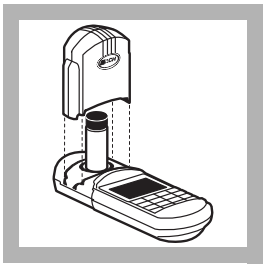
A five-minute reaction period will begin.

Note: *A deposit will remain after the reagent dissolves and will not affect results.*

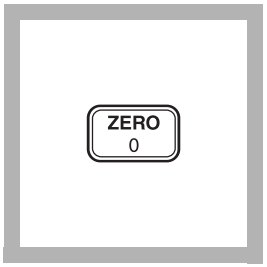
Note: *An amber color will develop if nitrate nitrogen is present.*



6. Fill a sample cell with at least 10 mL of sample (the blank).



7. When the timer beeps, place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

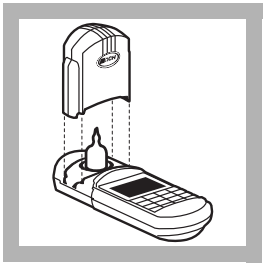


8. Press: **ZERO**

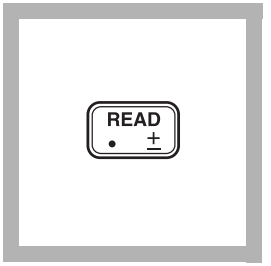
The cursor will move to the right, then the display will show:

0.0 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. Place the AccuVac Ampul into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L NO_3-N (or alternate form) will be displayed.

Note: *Use of the Standard Adjust feature for each new lot of reagent is highly recommended. See* Accuracy Check.

Note: *See Pollution Prevention and Waste Management for proper disposal of cadmium.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions* (*Section 1*) for more information.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL mixing cylinders with 25 mL of sample.

b) Snap the neck off a Nitrate Nitrogen Ampule Standard, 500 mg/L nitrate nitrogen.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of Nitrate Nitrogen Standard Solution to the three samples. Stopper and mix thoroughly.

d) For AccuVac analysis, transfer the solutions to clean, dry 50-mL beakers. For analysis with powder pillows, transfer only 10 mL of solution to clean, dry sample cells.

e) Analyze each sample as described above. The nitrate nitrogen (NO_3^--N) concentration should increase 2.0 mg/L for each 0.1 mL of standard added.

f) If these increases do not occur, see *Standard Additions* (*Section 1*) for more information.

Standard Solution Method

Use a Hach Nitrate-Nitrogen Standard Solution, 10.0 mg/L NO_3^--N, listed under Optional Reagents as the sample and perform the procedure as described above.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the curve adjustment. See *Section 1, Standard Curve Adjustmen*t for more information. If you are using a reagent blank correction, the blank correction should be entered before the Standard Adjust value is entered.

Method Performance

Precision

In a single laboratory using standard solutions of 25.0 mg/L nitrate nitrogen (NO_3^--N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of $\pm$0.3 mg/L nitrate nitrogen for program #50 and ±1.7 mg/L nitrate nitrogen for program # 51.

Estimated Detection Limit

The estimated detection limit for program 50 is 0.5 mg/L NO_3^--N and 0.8 mg/L NO_3^--N for program 51. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	All levels
Nitrite	All levels Compensate for nitrite interference as follows: Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. Add one drop of 30-g/L Phenol Solution to destroy the color. Proceed with Step 4. Report the results as total nitrate and nitrite.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels.

Summary Of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to gentisic acid to form an amber-colored product.

Pollution Prevention and Waste Management

NitraVer 5 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous wastes by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
NitraVer 5 Nitrate Reagent Powder Pillows	1 pillow	100/pkg	21061-69
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

REQUIRED REAGENTS (Using AccuVac Ampuls)

NitraVer 5 Nitrate Reagent AccuVac Ampul	1 ampul	25/pkg	25110-25

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41H
Stopper	1	6/pkg	1731-06

OPTIONAL REAGENTS

Bromine Water 30 g/L		29 mL*	2211-20
Nitrate Nitrogen Standard Solution, 10.0 mg/L as (NO_3^--N)		500 mL	307-49
Nitrate Nitrogen Standard Solution, 1000 mg/L as (NO_3^--N)		500 mL	12792-49
Nitrate Nitrogen Standard Solution, PourRite ampule, 500 mg/L as NO_3^--N, 2 mL		20/pkg	14260-20
Phenol Solution		29 mL	2112-20
Sodium Hydroxide Standard Solution, 5.0 N		50 mL*	2450-26
Sulfuric Acid, ACS		500 mL*	979-49
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit		each	24052-00
Cylinder, graduated, mixing, 25 mL		each	1896-40
Dropper, for 29-mL bottle		each	2258-00
pH Indicator Paper, 1 to 11 pH		5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable, with electrode		each	51700-10
Pipet Filler, safety bulb		each	14651-00
Pipet, serological, 2 mL		each	532-36
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite Ampule Breaker		each	24846-00
Thermometer, –20 to 110 °C, non-mercury		each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A. call 800-227-4224

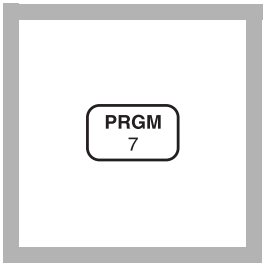
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes.

NITRATE, Mid Range (0 to 5.0 mg/L NO_3^--N) For water, wastewater and seawater*

Cadmium Reduction Method (Using Powder Pillows or AccuVac Ampuls)

Using Powder Pillows



1. Enter the stored program number for medium range nitrate nitrogen using powder pillows.

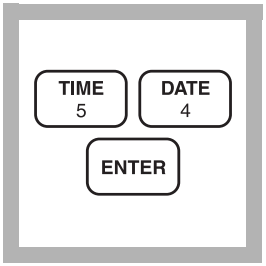
Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

Note: *Adjust the pH of stored samples before analysis.*

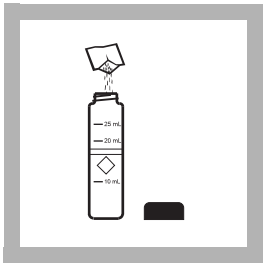


2. Press: **54 ENTER**

The display will show **mg/L, NO3-N** and the **ZERO** icon**.**

Note: *For alternate form (NO_3), press the* **CONC** *key.*

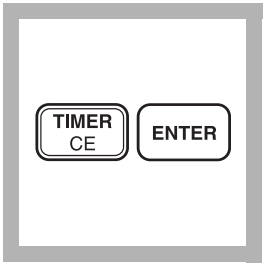
3. Fill two sample cells with 10 mL of sample each. One cell will be the prepared sample, the other is the blank. Set the blank aside.



4. Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow to one cell (the prepared sample). Cap the cell.

Note: *It is necessary to remove all the powder from the foil pouch by tapping repeatedly until no more powder comes out.*

* Seawater requires a manual calibration; see *Interferences*.

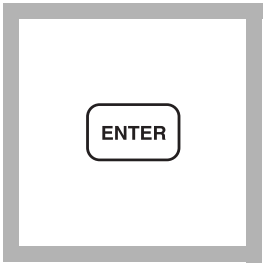


5. Press:

TIMER ENTER

A one-minute reaction period will begin. Shake the sample vigorously until the timer beeps.

Note: *Shaking time and technique influence color development. Low results usually occur if shaking is not vigorous enough. For most accurate results, do successive tests on a standard solution and adjust the shaking time by ±1 minute to obtain the correct result. See the Accuracy Check section for more information.*



6. After the timer beeps, the display will show:
5:00 TIMER 2

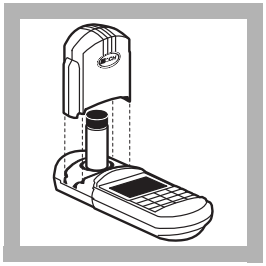
Press: **ENTER**

A five-minute reaction period will begin.

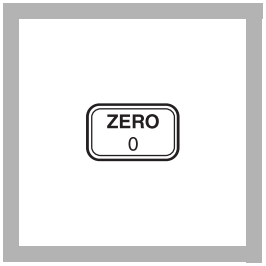
Note: *A cadmium deposit will remain after the NitraVer 5 Nitrate Reagent Powder dissolves and will not affect test results.*

Note: *An amber color will develop if nitrate nitrogen is present.*

7. After the timer beeps, wipe off any liquid or fingerprints.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

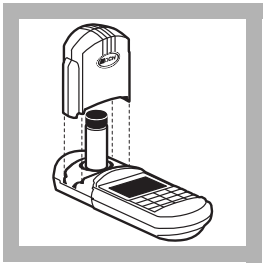


9. Press: **ZERO**

The cursor will move to the right, then the display will show:

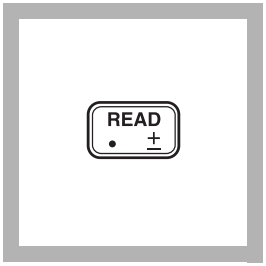
0.0 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit".*



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Read the sample within two minutes after the timer beeps.*



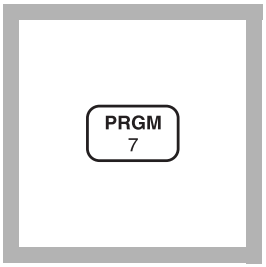
11. Press: **READ**

The cursor will move to the right, then the result in mg/L NO_3-N (or NO_3) will be displayed.

Note: *Use of the standard adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Note: *Rinse the sample cell immediately after use to remove all the cadmium particles. See Pollution Prevention and Waste Management following these steps for disposal of cadmium particles.*

Using AccuVac Ampuls



1. Enter the stored program number for medium range nitrate nitrogen using AccuVac Ampuls.

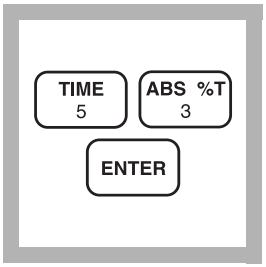
Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

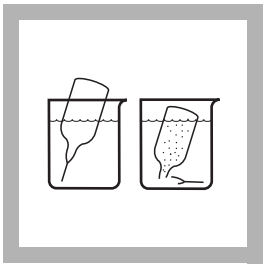
Note: *Adjust the pH of stored samples before analysis.*



2. Press: **53 ENTER**

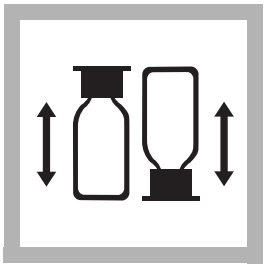
The display will show **mg/L, NO3-N** and the **ZERO** icon**.**

Note: *For alternate form (NO_3), press the* **CONC** *key.*



3. Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitraVer 5 Nitrate AccuVac Ampul with sample. Place a stopper over the tip of the ampul.

Note: *Keep the tip immersed while the ampul fills. The ampul will not fill completely to allow room for mixing.*

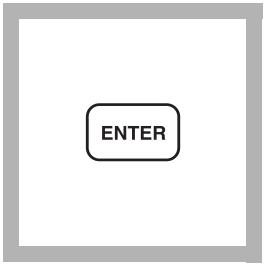


4. Press:

TIMER ENTER

A one-minute mixing period will begin. Invert the ampul repeatedly back and forth until the timer beeps. Wipe off any liquid or fingerprints after mixing.

Note: *Mixing speed and technique influence color development. For most accurate results, do successive tests on a standard solution and increase or decrease the mixing time to obtain the correct result. See Accuracy Check for more information.*



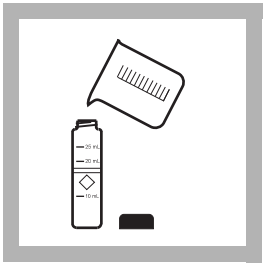
5. After the timer beeps, the display will show:
05:00 Timer 2

Press: **ENTER**

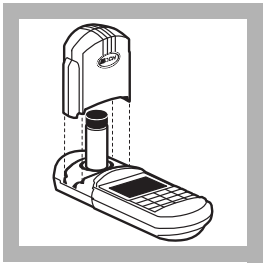
A five-minute reaction period will begin.

Note: *A cadmium deposit will remain after the NitraVer 5 Nitrate Reagent Powder dissolves and will not affect results.*

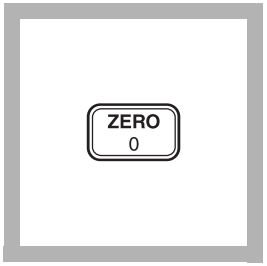
Note: *An amber color will develop if nitrate nitrogen is present.*



6. Fill a sample cell with at least 10 mL of sample (the blank).



7. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

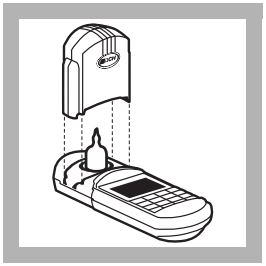


8. Press: **ZERO**

The cursor will move to the right, then the display will show:

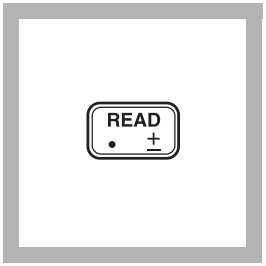
0.0 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. Place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Read the sample within two minutes after the timer beeps.*



10. Press: **READ**

The cursor will move to the right, then the result in mg/L NO_3-N (or NO_3) will be displayed.

Note: *Use of the standard adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions*, (*Section 1)* for more information.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Nitrate Nitrogen Ampule Standard Solution, 100 mg/L NO_3^--N.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the standard to the three samples. Stopper and mix well.

d) For analysis with AccuVac Ampuls, transfer the solutions to dry, clean 50 mL beakers. For analysis with powder pillows, transfer only 10 mL of the solution to dry, clean sample cells.

e) Analyze each sample as described above. The nitrate nitrogen (NO_3^--N) concentration should increase 0.4 mg/L for each 0.1 mL of standard added.

f) If these increases do not occur, see *Standard Additions* (*Section 1*) for more information.

Standard Solution Method

A 1.0 mg/L Nitrate Nitrogen Standard Solution is available from Hach. Use this standard in place of sample in the above procedure.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 1.00-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **1.0** to edit the

standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment . See *Section 1*, *Standard Curve Adjustmen*t for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 3.0 mg/L nitrate nitrogen (NO_3^--N) and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of ±0.2 mg/L nitrate nitrogen.

In a single laboratory using a standard solution of 3.0 mg/L NO_3^--N and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.1 mg/L nitrate nitrogen.

Estimated Detection Limit

The estimated detection limit for programs 53 and 54 is 0.2 mg/L NO_3-N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatments

Interfering Substance	Interference Levels and Treatments
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	All levels
Nitrite	All levels interfere. Compensate for nitrite interference as follows: **1.** Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. **2.** Add one drop of 30-g/L Phenol Solution to destroy the color. **3.** Proceed with Step 3. Report the results as total nitrate and nitrite.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels.

Summary of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to gentisic acid to form an amber-colored product.

Pollution Prevention and Waste Management

NitraVer 5 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Qty/ Test	Unit	Cat. No.
NitraVer 5 Nitrate Reagent Powder Pillows	1 pillow	100/pkg	21061-69
Sample Cell, 10-20-25 mL, w/ caps	2	6/pkg	24019-06

REQUIRED REAGENTS (Using AccuVac Ampuls)

NitraVer 5 Nitrate Reagent AccuVac Ampul	1 ampul	25/pkg	25110-25

REQUIRED APPARATUS (Using AccuVac Ampuls)

Beaker, 50 mL	1	each	500-41
Stopper	1	6/pkg	1731-06

OPTIONAL REAGENTS

Bromine Water 30 g/L		29 mL*	2211-20
Drinking Water Standard, Inorganics, (Fe^{-}, NO_3^{-}, SO_4^{2-}, PO_4^{3-})		500 mL	28330-49
Nitrate Nitrogen Standard Solution, 1.0 mg/L as NO_3^{-}-N		500 mL	2046-49
Nitrate Nitrogen Standard Solution, 100 mg/L as NO_3^{-}-N		500 mL	1947-49
Nitrate Nitrogen Standard Solution, PourRite Ampule, 100 mg/L as NO_3^{-}-N, 2 mL		20/pkg	1947-20
Phenol Solution, 30 g/L		29 mL	2112-20
Sodium Hydroxide Standard Solution, 5.0 N		50 mL SCDB*	2450-26
Sulfuric Acid, ACS		500 mL*	979-49
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

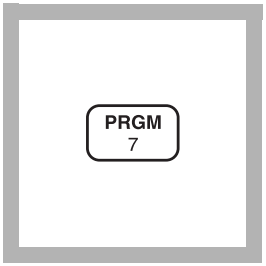
AccuVac Snapper Kit		each	24052-00
Cylinder, graduated, mixing, 25 mL		each	20886-40
Dropper, for 1-oz bottle		each	2258-00
pH Paper, 1 to 11 pH units		5 rolls/pkg	391-33
pH Meter, *sens**ion***™***1**, portable, with electrode		each	51700-10
Pipet Filler, safety bulb		each	14651-00
Pipet, serological, 2 mL		each	532-36
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet		50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite Ampule Breaker		each	24846-00

* Contact Hach for larger sizes.

NITRATE, Low Range (0 to 0.50 mg/L NO_3^--N)

For water, wastewater and seawater*

Cadmium Reduction Method



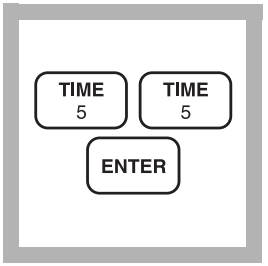
1. Enter the stored program number for low range nitrate nitrogen (NO_3^--N).

Press: **PRGM**

The display will show:

PRGM ?

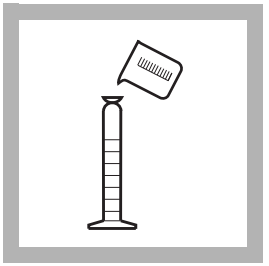
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **55 ENTER**

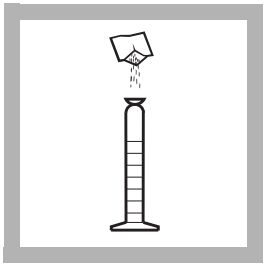
The display will show **mg/L, NO3-N** and the **ZERO** icon**.**

Note: *For alternate forms (NO_3), press the* **CONC** *key.*



3. Fill a 25-mL graduated mixing cylinder to the 15-mL mark with sample.

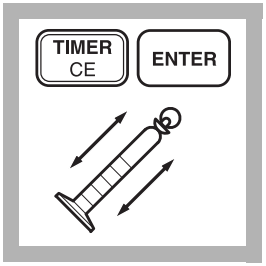
Note: *Adjust the pH of stored samples before analysis.*



4. Add the contents of one NitraVer 6 Nitrate Reagent Powder Pillow to the cylinder. Stopper.

Note: *It is necessary to remove* ***all*** *the powder from the foil pillow. Tap the pillow until no more powder pours out. Be sure to remove powder from the corners of the pillow.*

* Seawater requires a manual calibration; see Interferences.

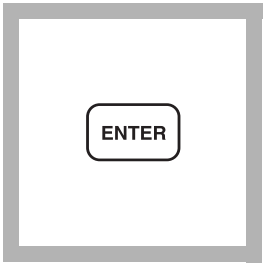


5. Press:

TIMER ENTER

A 3-minute reaction period will begin. Shake the cylinder vigorously throughout this three minute period.

Note: *Shaking time and technique influence color development. For most accurate results, analyze a standard solution several times and adjust the shaking time to obtain the correct result.*

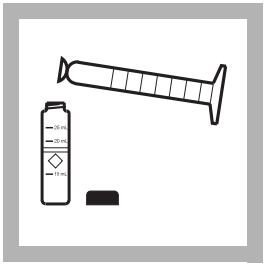


6. When the timer beeps, the display will show: **2:00 TIMER 2**

Press: **ENTER**

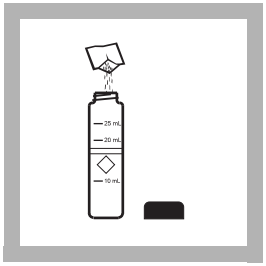
A 2-minute reaction period will begin.

Note: *A deposit will remain after the powder dissolves and will not affect results.*



7. When the timer beeps, pour 10 mL of the sample into a sample cell.

Note: *Do not transfer any cadmium particles.*



8. Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow to the sample cell (the prepared sample). Cap the cell and shake gently for 30 seconds.

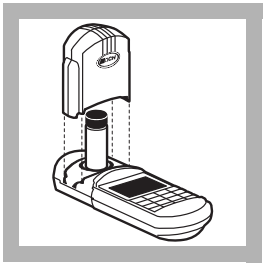
Note: *A pink color will form if nitrate is present.*

9. The display will show: **15:00 TIMER 3**

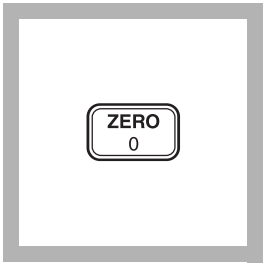
Press: **ENTER**

A 15-minute reaction period will begin.

Fill another sample cell (the blank) with 10 mL of sample.



10. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

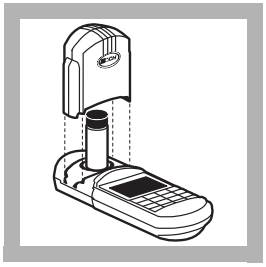


11. Press: **ZERO**

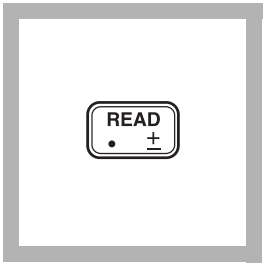
The cursor will move to the right, then the display will show:

0.00 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



12. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L NO_3^--N (or alternate form) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Note: *Rinse the sample cell and cylinder immediately after use to remove all cadmium particles.*

Note: *See Pollution Prevention and Waste Management for proper disposal of cadmium.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods, adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Do not use mercury compounds as preservatives. Correct the test result for volume additions; see *Correction for Volume Additions* (*Section 1*) for more information.

Accuracy Check

Standard additions Method

a) Fill three 25-mL graduated mixing cylinders with 15 mL of sample.

b) Snap the neck off a Nitrate Nitrogen Ampule Standard Solution, 12.0 mg/L NO_3^--N.

c) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of the standard to the three samples. Stopper and mix well.

d) Analyze each sample as described above. The nitrate nitrogen concentration should increase 0.08 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

Standard Solution Method

Prepare a 0.20 mg/L nitrate nitrogen standard by diluting 2.00 mL of a
10.0 mg/L Nitrate Nitrogen Standard Solution to 100.0 mL with deionized water. Use this standard in place of sample in Step 3.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 0.20-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **0.20** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the curve adjustment. If you are using a reagent blank correction, the blank correction should be entered before the Standard Adjust feature is entered. See *Section 1*, *Standard Curve Adjustmen*t for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 0.25 mg/L nitrate nitrogen (NO_3^--N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of $\pm$0.03 mg/L nitrate nitrogen.

Estimated Detection Limit

The estimated detection limit for program 55 is 0.01 mg/L NO_3^--N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Calcium	100 mg/L
Chloride	Chloride concentrations above 100 mg/L will cause low results. The test may be used at high chloride concentrations (seawater) but a calibration must be done using standards spiked to the same chloride concentration.
Ferric iron	All levels
Nitrite	All levels: This method measures both the nitrate and nitrite in the sample. If nitrite is present, the nitrite nitrogen test Program 60 should be done on the sample. Pretreat the nitrate nitrogen sample with the following pretreatment. Then subtract the amount of nitrite found from the results of the LR nitrate nitrogen test using the pretreated sample. **1.** Add 30-g/L Bromine Water dropwise to the sample in Step 3 until a yellow color remains. Mix after each drop. **2.** Add one drop of 30-g/L Phenol Solution to destroy the yellow color. **3.** Proceed with the LR Nitrate procedure.
pH	Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.
Strong oxidizing and reducing substances	Interfere at all levels

Summary of Method

Cadmium metal reduces nitrates present in the sample to nitrite. The nitrite ion reacts in an acidic medium with sulfanilic acid to form an intermediate diazonium salt which couples to chromotropic acid to form a pink-colored product.

Pollution Prevention and Waste Management

NitaVer 6 contains cadmium metal. Both samples and reagent blanks will contain cadmium (D006) at a concentration regulated as hazardous wastes by the Federal RCRA. Do not pour these solutions down the drain. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS

Low Range Nitrate Reagent Set (100 tests) 24298-00
Includes: (1) 21071-69, (1) 21072-49

Description	Quantity Required Per Test	Unit	Cat. No.
NitriVer 3 Nitrite Reagent Powder Pillows	1 pillow	100/pkg	21071-69
NitraVer 6 Nitrate Reagent Powder Pillows	1 pillow	100/pkg	21072-49

REQUIRED APPARATUS

Cylinder, graduated, mixing, 25 mL	1	each	1896-40
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Bromine Water, 30 g/L	29 mL*	2211-20
Nitrate Nitrogen Standard Solution, 10.0 mg/L as NO_3^--N	500 mL	307-49
Nitrate Nitrogen Standard Solution, Voluette ampule, 12 mg/L as NO_3^--N, 10 mL	16/pkg	14333-10
Phenol Solution, 30 g/L	29 mL	2112-20
Pretreatment Kit, contains: (1) 2112-20, (1) 2211-20	each	2268-00
Sodium Hydroxide Standard Solution, 5.0 N	50 mL* SCDB	2450-26
Sulfuric Acid, ACS	500 mL*	979-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker	each	21968-00
Dropper, for 29-mL bottle	each	2258-00
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH	5-roll/pkg	391-33
pH Meter, *sens**ion**™1*, portable, with electrode	each	51700-10
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet Filler, safety bulb	each	14651-00
Thermometer, –20 to 110 °C	each	26357-02

Nitrate at these levels can also be determined directly using the Nitrate Ion Selective Electrode (Cat. No. 23488-00).

For Technical Assistance, Price and Ordering

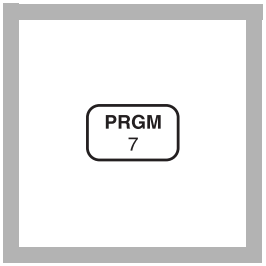
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes

NITRATE, High Range, Test 'N Tube (0 to 30.0 mg/L NO_3^--N)

Chromotropic Acid Method

For water and wastewater



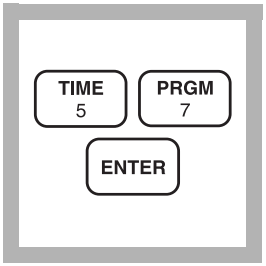
1. Enter the stored program number for Test 'N Tube nitrate nitrogen (NO_3^--N).

Press: **PRGM**

The display will show:

PRGM ?

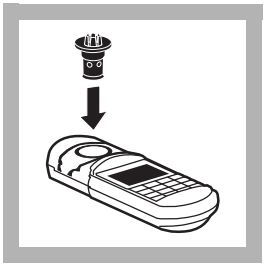
Note: *If samples cannot be analyzed immediately, see Sampling and Storage on page 331.*



2. Press: **57 ENTER**

The display will show **mg/L, NO3-N** and the **ZERO** icon**.**

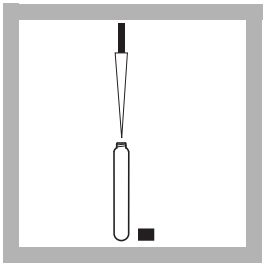
Note: *For alternate forms (NO_3) press the* **CONC** *key.*



3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

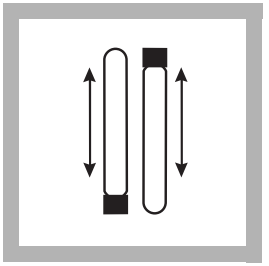
Note: *For proof of accuracy, use a 20 mg/L NO_3^--N standard in place of the sample.*

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



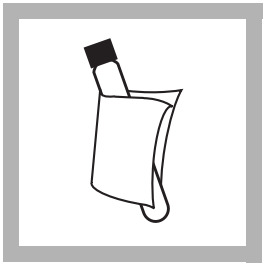
4. Remove the cap from a Nitrate Pretreatment Solution Vial and add 1 mL of sample (the blank).

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



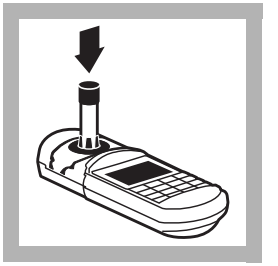
5. Cap the tube and invert 10 times to mix.

Note: *This test is technique-sensitive. Low results may occur if these instructions are not followed. Hold the vial vertical with the cap up. Invert the vial so the cap points down. Wait for all of the solution to flow to the cap end. Pause. Return the vial to the upright position. Wait for all the solution to flow to the vial bottom. This process equals 1 inversion. Do this 10 times.*



6. Clean the outside of the vial with a towel.

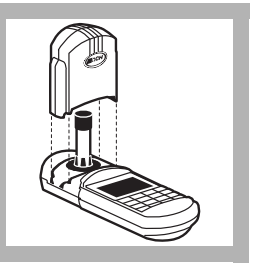
Note: *Wipe with a damp towel and follow with a dry one to remove fingerprints and other marks.*



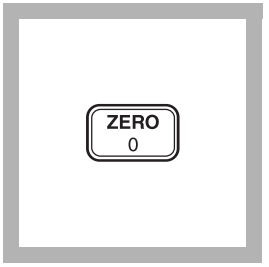
7. Place the blank in the vial adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



8. Cover the vial tightly with the instrument cap.

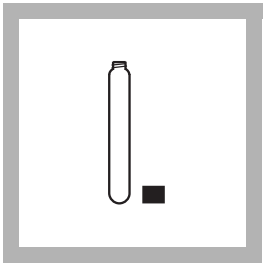


9. Press: **ZERO**

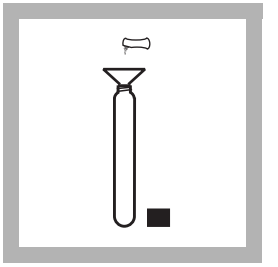
The cursor will move to the right, then the display will show:

0.0 mg/L NO3-N

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



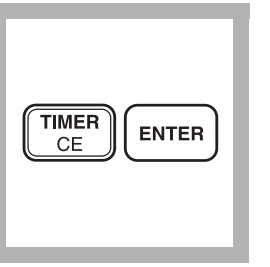
10. Remove the vial from the instrument. Remove the cap from the vial.



11. Using a funnel, add the contents of one NitraVer X Reagent B Powder Pillow to the vial. Cap. Invert 10 times to mix (this will be the prepared sample).

Note: *See Step 5 for inversion instructions*

Note: *Some solid matter will not dissolve.*



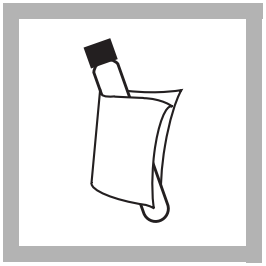
12. Press:

TIMER ENTER

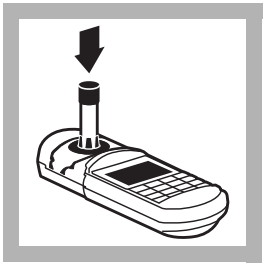
A five-minute reaction period will begin. Do not invert the vial again.

Note: *A yellow color will develop if nitrate nitrogen is present.*

Note: *Complete Steps 13-16 within five minutes after the timer beeps.*



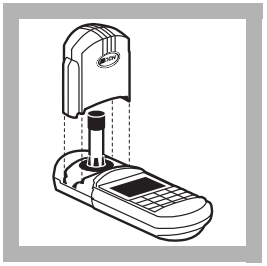
13. After the timer beeps, clean the outside of the vial with a damp towel and follow with a dry one to remove fingerprints and other marks.



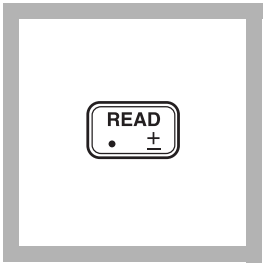
14. Place the prepared sample in the adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



15. Cover the vial tightly with the instrument cap.



16. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrate nitrogen (NO_3-N) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Store at 4 °C (39 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test. For longer storage periods (up to 14 days), adjust sample pH to 2 or less with sulfuric acid, ACS (about 2 mL per liter). Sample refrigeration is still required.

Before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution.

Do not use mercury compounds as preservatives.

Correct the test result for volume additions; see *Correction for Volume Additions* in *Section 1* for more information.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a fresh High Range Nitrate Nitrogen Voluette Ampule Standard, 500 mg/L NO_3^--N.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to the three mixing cylinders, respectively. Mix each thoroughly.

d) Analyze each sample as described in the procedure; use a 1-mL aliquot of the spiked sample in each test. The nitrogen concentration should increase 2.0 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* (*Section 1*) for more information.

Standard Solution Method

To test accuracy, prepare a 20.0 mg/L nitrate nitrogen standard solution by pipetting 2.00 mL of a High Range Nitrate Nitrogen Voluette Ampule Standard Solution, 500 mg/L NO_3^--N, into a 50 mL Class A volumetric flask. Dilute to the line with deionized water. Substitute this standard for the sample and perform the test as described in the procedure.

Method Performance

Precision

In a single laboratory, using a standard solution of 25.0 mg/L nitrate nitrogen (NO_3^--N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ± 0.5 mg/L NO_3-N.

Estimated Detection Limit

The estimated detection limit for program 57 is 0.3 mg/L NO_3-N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level
Barium	A negative interference at concentrations greater than 1 mg/L.
Chloride	Does not interfere below 1000 mg/L.
Hardness	Does not interfere.
Nitrite	A positive interference at concentrations greater than 12 mg/L. Remove nitrite interference up to 100 mg/L by adding 400 mg of urea (one full 0.5 g Hach measuring spoon) to 10 mL of sample. Swirl to dissolve. Proceed with the nitrate test as usual.

Summary of Method

Nitrate in the sample reacts with chromotropic acid under strongly acidic conditions to yield a yellow product with a maximum absorbance at 410 nm.

REQUIRED REAGENTS

	Cat. No.
NitraVer X Nitrate, High Range Test 'N Tube Reagent Set (50 tests)	26053-45
Includes: (1) 26055-46, (1) 272-42, *(50) Nitrate Pretreatment Solution Vials	

Description	Quantity Required Per Test	Unit	Cat. No.
Nitrate Pretreatment Solution Vials	1	50/pkg	*
NitraVer X Reagent B Powder Pillows	1	50/pkg	26055-46

REQUIRED APPARATUS

COD Vial Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	varies	50/pkg	21856-96
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS

Nitrate-Nitrogen Standard Solution, Voluette Ampules, 500 mg/L N	16/pkg	14260-10
Sodium Hydroxide Standard Solution, 5.0 N	50 mL	2450-26
Sulfuric Acid, ACS, concentrated	500 mL	979-49
Urea, ACS	100 g	11237-26
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Cylinder, graduated, mixing, 25-mL (3 required)	each	26363-40
Flask, volumetric, Class A, 50 mL	each	14574-41
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
Pipet, volumetric, Class A, 2 mL	each	14515-36
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Spoon, measuring, 0.5 g	each	907-00

For Technical Assistance, Price and Ordering

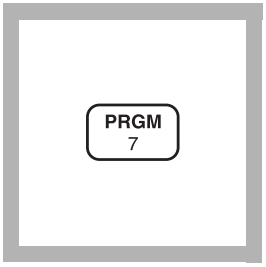
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Not available separately.

NITRITE, High Range (0 to 150 mg/L NO_2^-)

Ferrous Sulfate Method*



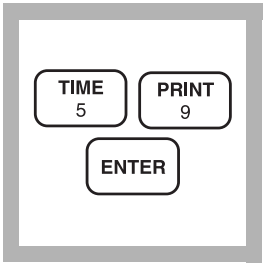
1. Enter the stored program number for high range nitrite (NO_2^-).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

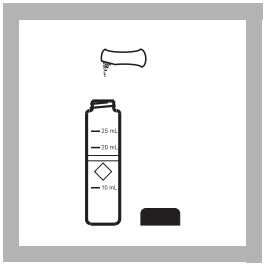


2. Press: **59 ENTER**

The display will show **mg/L, NO2** and the **ZERO** icon**.**

Note: *For alternate forms (NO_2-N, $NaNO_2$), press the* **CONC** *key.*

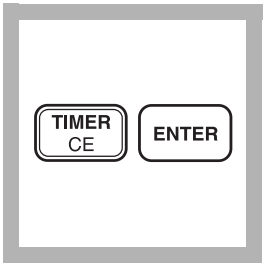
3. Fill a sample cell with 10 mL of sample.



4. Add the contents of one NitriVer 2 Nitrite Reagent Powder Pillow. Cap the cell and invert 5-7 times to mix (the prepared sample).

Note: *A greenish-brown color will develop if nitrite is present.*

Note: *Avoid excessive mixing or low results may occur. Accuracy is not affected by undissolved powder.*

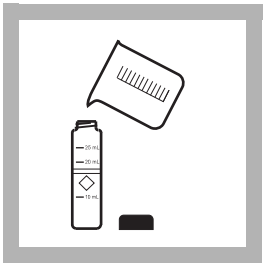


5. Press:

TIMER ENTER

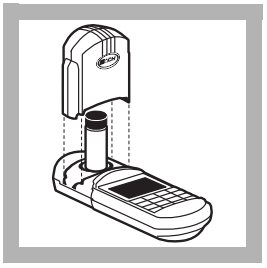
A ten-minute reaction period will begin.

Do not move or disturb the sample cell during this reaction period.

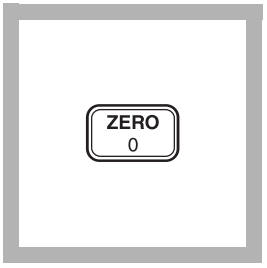


6. Fill another sample cell with 10 mL of sample (the blank). Clean the outside of the cells with a towel.

Note: *Wiping with a damp towel, followed by a dry one, removes fingerprints and other marks.*



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



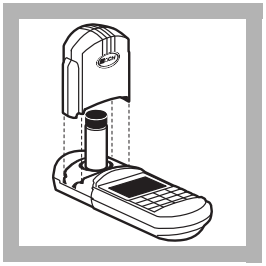
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L NO2

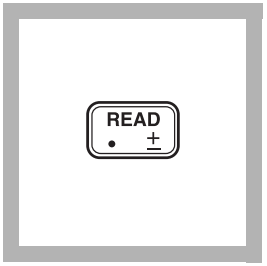
Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*

* Adapted from McAlpine, R. and Soule, B., *Qualitative Chemical Analysis*, New York, 476,575 (1933)



9. After the timer beeps, gently invert the prepared sample twice. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Avoid excessive mixing or low results may occur.*



10. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrite will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. If prompt analysis is impossible, store at 4 °C (39 °F) or lower if the sample is to be analyzed within 48 hours. Warm to room temperature before running the test. Do not use acid preservatives. Remove suspended solids by filtration.

Accuracy Check

Standard Solution Method

Dissolve 0.150 grams of fresh sodium nitrite and dilute to 1000 mL with deionized water to prepare a 100 mg/L nitrite standard solution. Prepare this solution daily.

Alternatively, make a dilution of a fresh Hach Nitrite Standard Solution, 821 mg/L NO_2^- (250 mg/L NO_2^--N) using Class A glassware. Dilute 10 mL of this standard to 100 mL with deionized water to give an
82 mg/L nitrite standard. Prepare this solution just before use. Using this solution as the sample, perform the nitrite procedure as described above.

Method Performance

Precision

In a single laboratory using a standard solution of 123 mg/L nitrite and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of ±1 mg/L nitrite.

Estimated Detection Limit

The estimated detection limit for program 59 is 2 mg/L NO_2^-. For more information on the estimated detection limit, see *Section 1*.

Interferences

This test does not measure nitrates nor is it applicable to glycol based samples. Dilute glycol based samples and follow the Low Range Nitrite Procedure.

Summary of Method

The method uses ferrous sulfate in an acidic medium to reduce nitrite to nitrous oxide. Ferrous ions combine with the nitrous oxide to form a greenish-brown complex in direct proportion to the nitrite present.

REQUIRED REAGENTS AND APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
NitriVer 2 Nitrite Reagent Powder Pillows	1 pillow	100/pkg	21075-69
Sample cell, 10-20-25, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Nitrite Standard Solution, 821 mg/L NO_2 (250 mg/L NO_2^--N)		500 mL	23402-49
Sodium Nitrite, ACS		454 g	2452-01
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Balance, analytical, 110 V, Acculab UI Series, 120 g		each	26947-00
Flask, volumetric, 1000 mL		each	14547-53
Flask, volumetric, 100 mL, Class A		each	14574-42
Pipet, volumetric, 10.00 mL, Class A		each	14515-38
Pipet Filler, safety bulb		each	14651-00

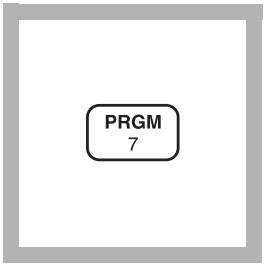
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITRITE, Low Range (0 to 0.350 mg/L NO_2^--N) For water, wastewater, seawater

Diazotization Method* (Powder Pillows or AccuVac Ampuls);
USEPA approved for reporting wastewater and drinking water analyses.



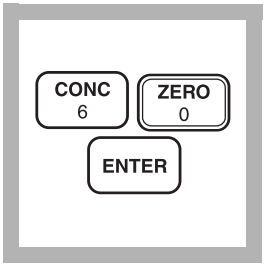
1. Enter the stored program number for nitrite nitrogen (NO_2^--N), powder pillows.

Press: **PRGM**

The display will show:

PRGM ?

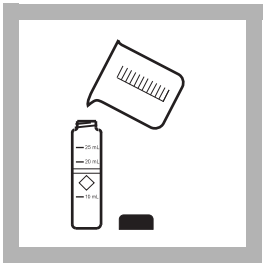
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



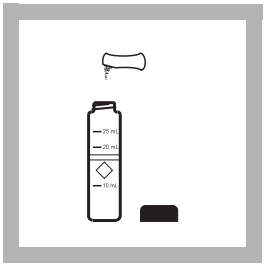
2. Press: **60 ENTER**

The display will show **mg/L, NO2-N** and the **ZERO** icon.

Note: *For alternate forms (NO_2^-, $NaNO_2$), press the* **CONC** *key.*



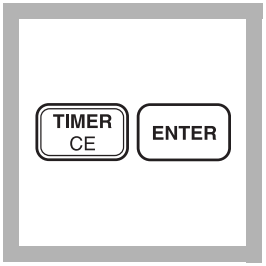
3. Fill a sample cell with 10 mL of sample.



4. Add the contents of one NitriVer 3 Nitrite Reagent Powder Pillow to the sample cell. Cap the cell and shake to dissolve.

Note: *Accuracy is not affected by undissolved powder.*

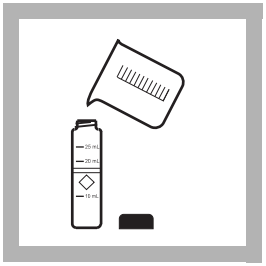
* *Federal Register*, 44(85) 25505 (May 1, 1979)



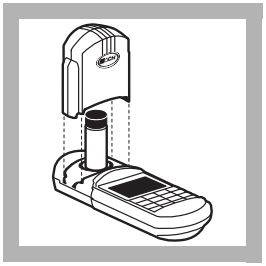
5. Press:
TIMER ENTER

A 15-minute reaction period will begin.

Note: *A pink color will develop if nitrite is present.*

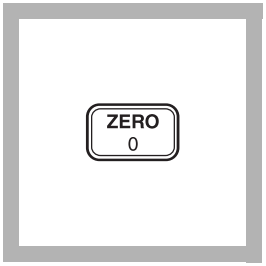


6. When the timer beeps, fill an empty sample cell with 10 mL of sample (the blank).



7. Wipe the outside of the sample cell with a towel. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Wiping with a damp cloth, followed by a dry pne, removes fingerprints and other marks.*

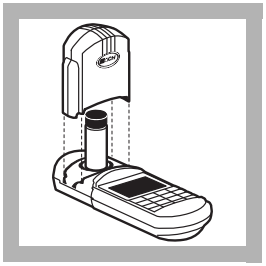


8. Press: **ZERO**

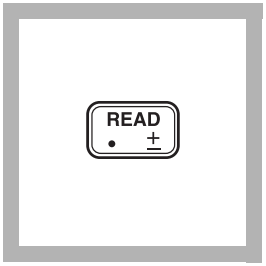
The cursor will move to the right, then the display will show:

0.000 mg/L NO2-N

Note: *If Reagent Blank Correction is on, the display may flash "limit." See Section 1.*



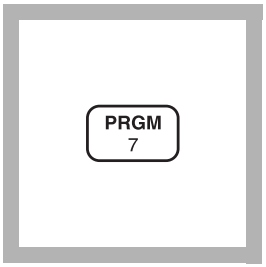
9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrite nitrogen (or an alternate form) will be displayed.

Using AccuVac Ampuls



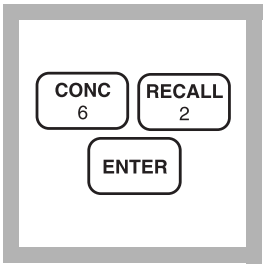
1. Enter the stored program number for nitrite nitrogen (NO_2^--N), AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

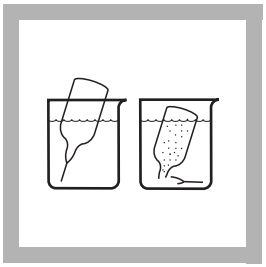
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **62 ENTER**

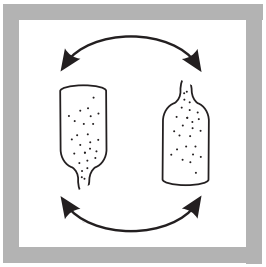
The display will show **mg/L, NO2-N** and the **ZERO** icon**.**

Note: *For alternate forms (NO_2^-, $NaNO_2$), press the* **CONC** *key.*



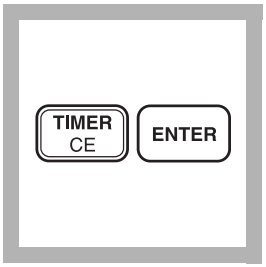
3. Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitriVer 3 Nitrite AccuVac Ampul with the sample.

Note: *Keep the tip immersed while the ampul fills completely.*



4. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

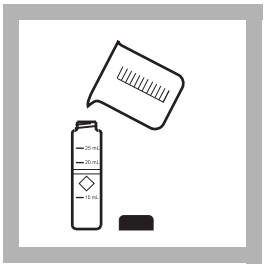
Note: *Accuracy is not affected by undissolved powder.*



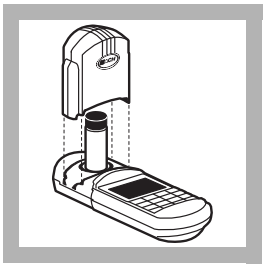
5. Press:
TIMER ENTER

A 15-minute reaction period will begin.

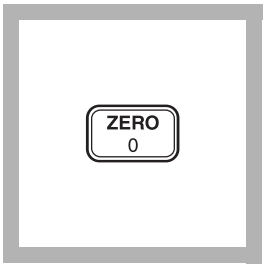
Note: *A pink color will develop if nitrite is present.*



6. When the timer beeps, fill a sample cell with at least 10 mL of sample (the blank).



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

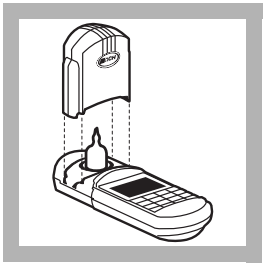


8. Press: **ZERO**

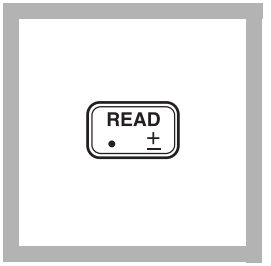
The cursor will move to the right, then the display will show:

0.000 mg/L NO2-N

Note: *If Reagent Blank Correction is on, the display may flash "limit." See Section 1.*



9. Place the AccuVac Ampul into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrite nitrogen will be displayed.

Sampling and Storage

Collect samples in clean plastic or glass bottles.

Store at 4 °C (39 °F) or lower and analyze within 48 hours. Warm to room temperature before running the test.

Do not use acid preservatives.

Remove the suspended solids by filtration.

Accuracy Check

Standard Solution Method

Pipet 5.00 mL of a fresh 250 mg/L NO_2^--N standard into a 250.0 mL volumetric flask. Dilute to the mark with deionized water. This makes a 5.00-mg/L intermediate standard. To prepare a 0.100-mg/L NO_2^--N standard solution, dilute 10.00 mL of the 5.00-mg/L intermediate standard to 500 mL in a volumetric flask. Prepare this solution immediately before use.

Run the test using the 0.100 mg/L NO_2^--N standard in place of the sample. Results should be between 0.090 and 0.110 mg/L NO_2^--N.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.250 mg/L nitrite nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.001 mg/L NO_2^--N for the powder pillow method and ±0.003 mg/L NO_2^--N for the
AccuVac method.

Estimated Detection Limit

The estimated detection limit for programs 60 and 62 is 0.005 mg/L NO_2^--N. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels
Antiminous ions	Interfere by causing precipitation
Auric ions	Interfere by causing precipitation
Bismuth ions	Interfere by causing precipitation
Chloroplatinate ions	Interfere by causing precipitation
Cupric ions	Cause low results
Ferric ions	Interfere by causing precipitation
Ferrous ions	Cause low results
Lead ions	Interfere by causing precipitation
Mercurous ions	Interfere by causing precipitation
Metavanadate ions	Interfere by causing precipitation
Nitrate	Very high levels of nitrate (>100 mg/L nitrate as N) appear to undergo a slight amount of reduction to nitrite, either spontaneously or during the course of the test. A small amount of nitrite will be found at these levels.
Silver ions	Interfere by causing precipitation
Strong oxidizing and reducing substances	Interfere at all levels

Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink colored complex directly proportional to the amount of nitrite present.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
NitriVer 3 Nitrite Reagent Powder Pillows	1 pillow	100/pkg	21071-69
or			
NitriVer 3 Nitrite Reagent AccuVac Ampuls	1 ampul	25/pkg	25120-25

REQUIRED APPARATUS

Beaker, 50 mL (for AccuVac procedure)	1	each	500-41H
or			
Sample Cells, 10-20-25 mL (powder pillow procedure)	2	6/pkg	24019-06

OPTIONAL REAGENTS

Nitrite Standard Solution, 250 mg/L as NO_2^--N		500 mL	23402-49
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Description	Unit	Cat. No.
AccuVac Snapper Kit	each	24052-00
Flask, volumetric, 250 mL	each	14574-46
Flask, volumetric, 500 mL	each	14574-49
Pipet, serological, 10 mL	each	532-38
Pipet, TenSette, 1 to 10 mL	each	19700-01
Pipet Tips for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Pipet Filler, safety bulb	each	14651-00
Thermometer, –20 to 110 °C	each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A. call 800-227-4224

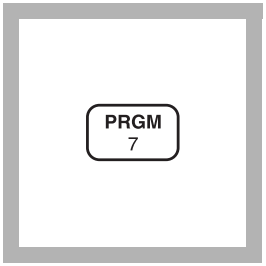
Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITRITE, Low Range, Test 'N Tube (0–0.500 mg/L NO_2^--N)

Diazotization Method

For water, wastewater, and seawater

USEPA approved for wastewater analysis*



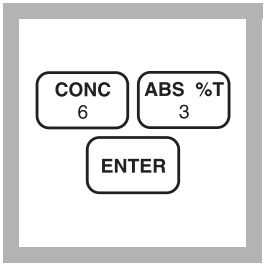
1. Enter the stored program number for nitrite nitrogen (NO_2^--N), Test 'N Tube.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



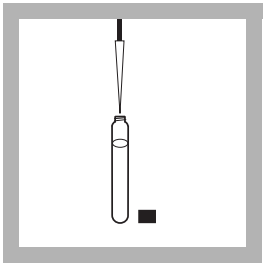
2. Press: **63 ENTER**

The display will show **mg/L, NO2-N** and the **ZERO** icon.

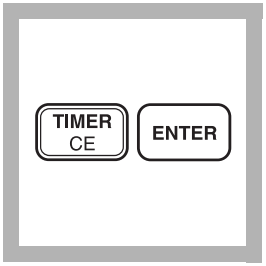
Note: *For alternate forms (NO_2^-, $NaNO_2$), press the* **CONC** *key.*

3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert.

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



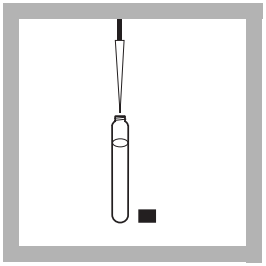
4. Fill a Test 'N Tube NitriVer® 3 Nitrite vial with 5 mL of sample. Cap and shake to dissolve powder. This is the prepared sample.



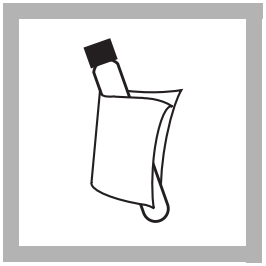
5. Press:
TIMER ENTER

A 20-minute reaction period will begin.

Note: *A pink color will develop if nitrite is present.*

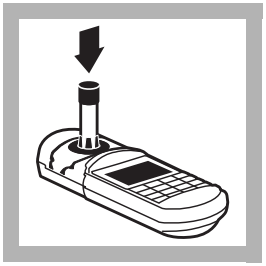


6. When the timer beeps, fill an empty Test 'N Tube vial with 5 mL of sample (the blank).



7. Clean the outside of the vials with a towel.

Note: *Wipe with a damp towel and follow with a dry one to remove fingerprints and other marks.*

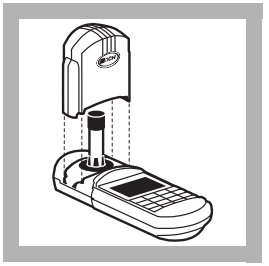


8. Place the blank in the vial adapter.

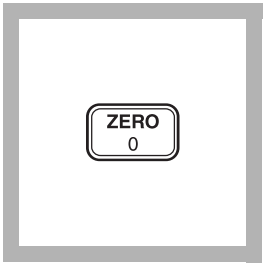
Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*

* *Federal Register,* 44(85) 25505 (May 1, 1979).



9. Cover the sample cell tightly with the instrument cap.

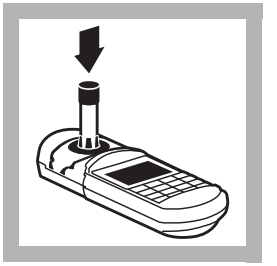


10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.000 mg/L NO2-N

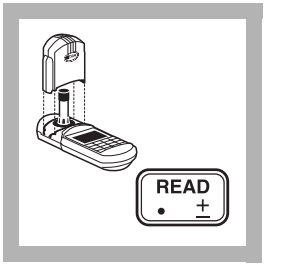
Note: *If the reagent blank correction is on, the display may flash "limit." See Section 1.*



11. Place the prepared sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Tightly cover the sample cell with the instrument cap.

Press: **READ**

The cursor will move to the right, then the result in mg/L nitrite nitrogen (or an alternate form) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles.

Store at 4 °C (39 °F) or lower and analyze within 48 hours. Warm to room temperature before running the test.

Do not use acid preservatives.

Remove suspended solids by filtration.

Accuracy Check

Standard Solution Method

Pipet 5.00 mL of a fresh Hach standard, 250 mg/L as NO_2^--N into a Class A 250-mL volumetric flask. Dilute to the line with deionized water to make a 5.00-mg/L intermediate standard. Pipet 10.00 mL of the 5.0-mg/L intermediate standard into a Class A 500-mL volumetric flask. Dilute to the line with deionized water to make a 0.100 mg/L NO_2^--N standard solution. Prepare immediately before use.

Run the test using the 0.100 mg/L NO_2^--N standard in place of the sample. Results should be between 0.090 and 0.110 mg/L NO_2^--N.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.250 mg/L nitrite nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.004 mg/L NO_2^--N.

Estimated Detection Limit

The estimated detection limit for program 63 is 0.006 mg/L NO_2^--N. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels
Antiminous ions	Interfere by causing precipitation
Auric ions	Interfere by causing precipitation
Bismuth ions	Interfere by causing precipitation
Chloroplatinate ions	Interfere by causing precipitation
Cupric ions	Cause low results
Ferric ions	Interfere by causing precipitation
Ferrous ions	Cause low results
Lead ions	Interfere by causing precipitation
Mercurous ions	Interfere by causing precipitation
Metavanadate ions	Interfere by causing precipitation
Nitrate	Very high levels of nitrate (>100 mg/L nitrate as N) appear to undergo a slight amount of reduction to nitrite, either spontaneously or during the course of the test. A small amount of nitrite will be found at these levels.
Silver ions	Interfere by causing precipitation
Strong oxidizing and reducing substances	Interfere at all levels

Summary of Method

Nitrite in the sample reacts with sulfanilic acid to form an intermediate diazonium salt. This couples with chromotropic acid to produce a pink-colored complex directly proportional to the amount of nitrite present.

REQUIRED REAGENTS

Description	Cat. No.
NitriVer® 3 Nitrite, Low Range Test 'N Tube Reagent Set (50 tests)	26083-45
Includes:	
(50) NitriVer® 3 Nitrite Test 'N Tube Vials	*
Vials, 6 x 100 mm, 6/pkg	22758-06
Caps, for 22758-06 vials, 6/pkg	22411-06
Deionized water, 100-mL	272-42

REQUIRED APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
COD/TNT Adapter	1	each	48464-00
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette, 1 to 10 mL	1	each	19700-10
Pipet Tips for 19700-10 TenSette Pipet	1	50/pkg	21997-96

OPTIONAL REAGENTS

Nitrite Standard Solution, 250 mg/L as NO_2^--N	500	mL	23402-49
Water, deionized	4	L	272-56

OPTIONAL APPARATUS

Flask, volumetric, 250 mL		each	14574-46
Flask, volumetric, 500 mL		each	14574-49
Pipet, volumetric, Class A, 10.00 mL		each	14515-38

For Technical Assistance, Price and Ordering

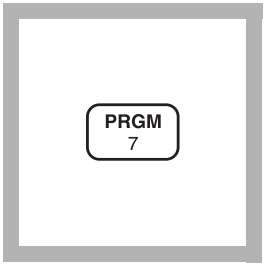
In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Not available separately.

NITROGEN, AMMONIA (0 to 0.50 mg/L NH_3-N) For water, wastewater, seawater

Salicylate Method*

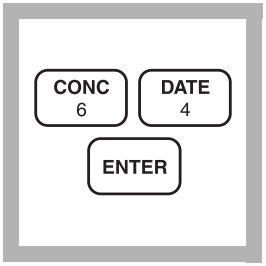


1. Enter the stored program number for ammonia nitrogen (NH_3-N).

Press: **PRGM**

The display will show:

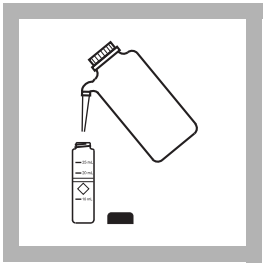
PRGM ?



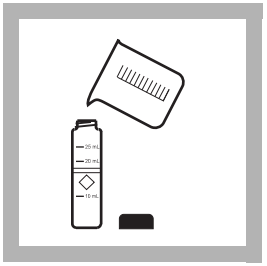
2. Press: 64 ENTER

The display will show **mg/L, NH3-N** and the **ZERO** icon**.**

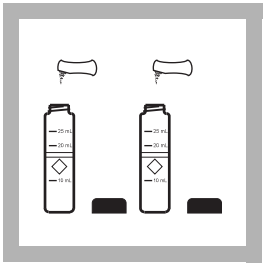
Note: *For alternate forms (NH_3, NH_4), press the* **CONC** *key.*



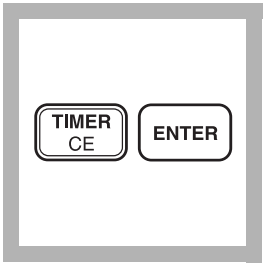
3. Fill a sample cell with 10 mL of deionized water (the blank).



4. Fill a second sample cell with 10 mL of the sample.



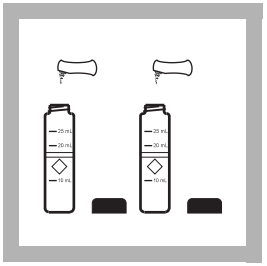
5. Add the contents of one Ammonia Salicylate Reagent Powder Pillow to each sample cell. Cap both cells and shake to dissolve.



6. Press:

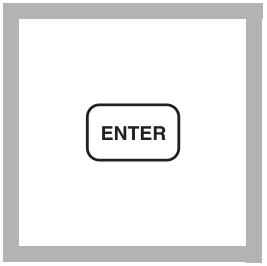
TIMER ENTER

A three-minute reaction period will begin.



7. After the timer beeps add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each sample cell. Cap the cells and shake to dissolve the reagent.

Note: *A green color will develop if ammonia nitrogen is present.*

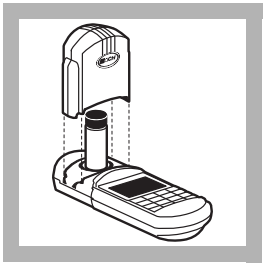


8. The display will show: **15:00 TIMER 2**

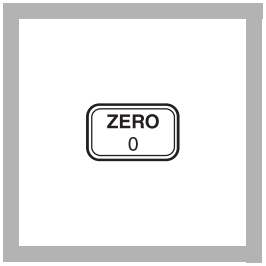
Press: **ENTER**

A 15-minute reaction period will begin.

* Adapted from Clin. Chim. Acta., 14 403 (1966)



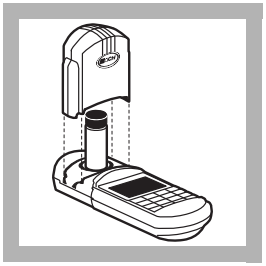
9. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



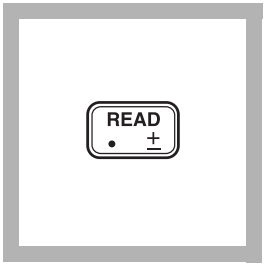
10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L NH3-N



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L ammonia nitrogen will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Most reliable results are obtained when samples are analyzed as soon as possible after collection.

If chlorine is known to be present, the sample must be treated immediately with sodium thiosulfate. Add one drop of Sodium Thiosulfate Standard Solution, 0.1 N, for each 0.3 mg of chlorine present in a one liter sample.

To preserve the sample, adjust the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter). Store samples at 4 °C or less. Samples preserved in this manner can be stored up to 28 days. Just before testing the stored sample, warm to room temperature and neutralize with 5.0 N Sodium Hydroxide Standard Solution. Correct the test result for volume additions; see *Correction for Volume Additions*, in *Section 1* for more detailed information.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL mixing cylinders with 20 mL of sample.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of Ammonium Nitrogen Standard, 10 mg/L as NH_3-N to the three samples. Stopper the cylinders and mix well.

c) Analyze a 10-mL portion of sample as described above. The ammonia nitrogen concentration should increase 0.05 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions (Section 1)* for more information.

Standard Solution Method

Prepare a 0.40 mg/L ammonia nitrogen standard by diluting 4.00 mL of the Ammonia Nitrogen Standard Solution, 10 mg/L, to 100 mL with deionized water. Or, using the TenSette Pipet, prepare a 0.40 mg/L ammonia nitrogen standard by diluting 0.8 mL of a Ammonia Nitrogen Voluette Standard Solution, 50 mg/L as NH_3-N, to 100 mL with
deionized water.

Method Performance

Precision

In a single laboratory using a standard solution of 0.40 mg/L ammonia nitrogen (NH_3-N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of $\pm$0.02 mg/L ammonia nitrogen.

Estimated Detection Limit

The estimated detection limit for program 64 is 0.02 mg/L NH_3-N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatments.

Interfering Substance	Interference Level and Treatments
Calcium	Greater than 1000 mg/L as $CaCO_3$
Glycine, hydrazine	Less common. Will cause intensified colors in the prepared sample.
Iron	All levels. Correct for iron interference as follows: **1.** Determine the amount of iron present in the sample using one of the Total Iron procedures. **2.** Prepare a deionized water sample containing the same iron concentration as the original sample. Run the procedure on this solution to determine the interference due to iron. Subtract this value from the result in Step 12 obtained on the original sample.
Magnesium	Greater than 6000 mg/L as $CaCO_3$
Nitrate	Greater than 100 mg/L as NO_3^--N
Nitrite	Greater than 12 mg/L as NO_2^--N
Phosphate	Greater than 100 mg/L as PO_4^{3-}-P
Sulfate	Greater than 300 mg/L as SO_4^{2-}
Sulfide	Sulfide will intensify the color. Eliminate sulfide interference as follows: **1.** Measure about 350 mL of sample in a 500-mL erlenmeyer flask. **2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix. **3.** Filter the sample through a folded filter paper. **4.** Use the filtered solution in Step 3.
Turbidity, sample color	Turbidity and sample color will give erroneous high values. Samples with severe interferences require distillation. Albuminoid nitrogen samples also require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set. See the Optional Apparatus list.

Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

REQUIRED REAGENTS AND APPARATUS

			Cat. No.
Ammonia Nitrogen Reagent Set for 10-mL samples (100 tests)			26680-00
Includes: (2) 26531-99, (2) 26532-99			
Description	**Quantity Required Per Test**	**Unit**	**Cat. No.**
Ammonia Cyanurate Reagent Powder Pillows	2 pillows	100/pkg	26531-99
Ammonia Salicylate Reagent Powder Pillows	2 pillows	100/pkg	26532-99
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Ammonia Nitrogen Standard Solution, 10 mg/L as NH_3-N	500 mL	153-49
Ammonia Nitrogen, PourRite Ampules, 50 mg/L as NH_3-N, 2 mL	20/pkg	14791-20
Cylinder, graduated, mixing, 25 mL	each	20886-40
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	1045-32
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB	2450-26
Sodium Thiosulfate Standard Solution, 0.1 N	100 mL MDB	323-32
Sulfide Inhibitor Reagent Powder Pillows	100/pkg	2418-99
Sulfuric Acid, concentrated, ACS	500 mL	979-49
Sulfuric Acid Standard Solution, 1.0 N	100 mL MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Cylinder, graduated, polypropylene, 500 mL	each	1081-49
Distillation Heater and Support Apparatus, 115 V	each	22744-00
Distillation Heater and Support Apparatus, 230 V	each	22744-02
Distillation Set, General Purpose	each	22653-00
Filter Paper, folded, 12.5 cm	100	1894-57
Flask, Erlenmeyer, polypropylene, 500 mL	each	1082-49
Flask, volumetric, Class A, 100 mL	each	14574-42
Funnel, poly, 65 mm	each	1083-67
pH Meter, *sens**ion**™1*, portable, with electrode	each	51700-10
Pipet Filler, safety bulb	each	14651-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 2.0 mL	each	14515-36
PourRite Ampule Breaker Kit	each	24846-00
Thermometer, –20 to 110 °C	each	26357-02

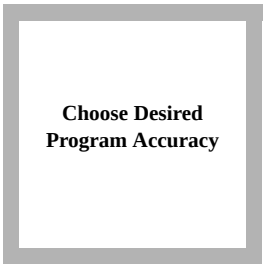
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

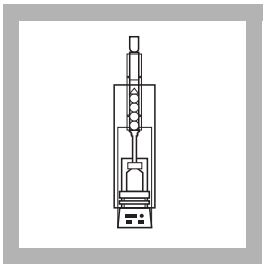
Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITROGEN, TOTAL KJELDAHL (0 to 150 mg/L)

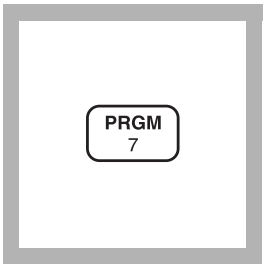
Nessler Method* (digestion required) **For water, wastewater and sludge**



1. A User-Entered Calibration is necessary to obtain the most accurate results. See the User Calibration section following these steps.



2. Digest the sample as described in the Digesdahl Apparatus Instruction manual. Digest an equal amount of deionized water as the blank.

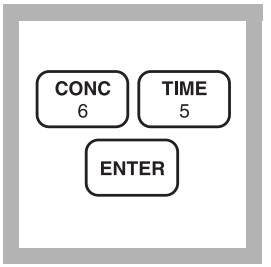


3. Enter the stored program number for total Kjeldahl nitrogen.

Press: **PRGM**

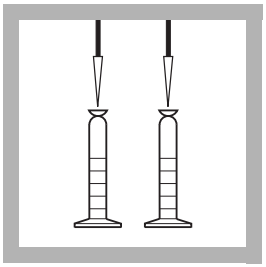
The display will show:

PRGM ?

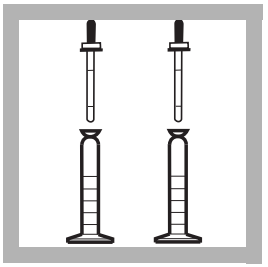


4. Press: **65 ENTER**

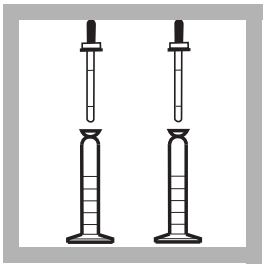
The display will show **mg/L, TKN** and the **ZERO** icon**.**



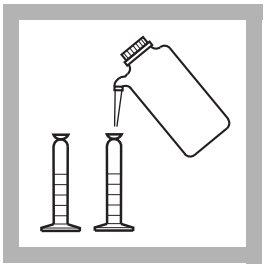
5. Select the appropriate analysis volume of the digested sample given in *Table 1* on page *357*. Pipet the analysis volume from the sample and the digested blank into separate 25-mL mixing graduated cylinders.



6. Add one drop of TKN Indicator to each cylinder. Add 8.0 N KOH dropwise to each cylinder, mixing after each addition. Continue until the first apparent blue color is visible.



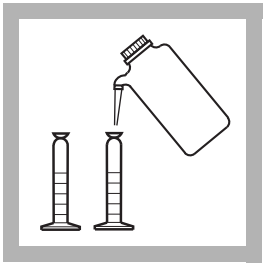
7. Add 1.0 N KOH to each cylinder, one drop at a time, mixing after each addition. Continue until the first permanent blue color appears.



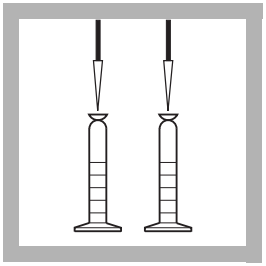
8. Fill both mixing cylinders to the 20-mL mark with deionized water. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix. Add 3 drops of Polyvinyl Alcohol Dispersing Agent to each cylinder. Invert several times to mix.

Note: *Hold the dropping bottles upright while dispensing.*

* Adapted from: Hach et al., *Journal of Association of Official Analytical Chemists,* 70 (5) 783-787 (1987); Hach et al., *Journal of Agricultural and Food Chemistry,* 33 (6) 1117-1123 (1985); *Standard Methods for the Examination of Water and Wastewater.*

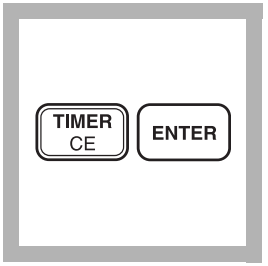


9. Fill both cylinders to the 25-mL mark with deionized water.



10. Pipet 1 mL of Nesslers Reagent to each cylinder. Stopper, invert repeatedly. The solution should not be hazy.

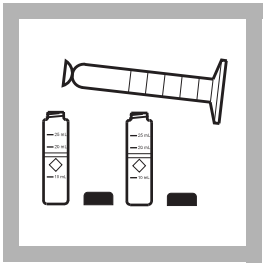
Note: *Any haze (turbidity) will cause incorrect results.*



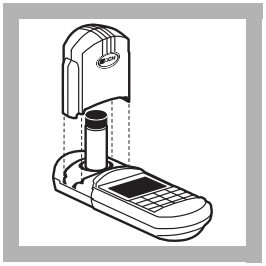
11. Press:

TIMER ENTER

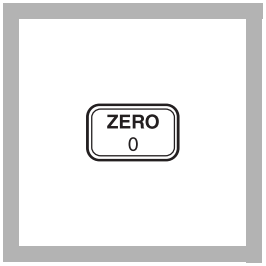
A two-minute reaction period will begin.



12. When the timer beeps, pour the contents of each cylinder into a separate labeled sample cell.



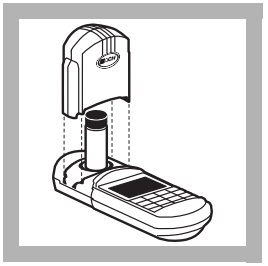
13. Place the blank into a cell holder. Tightly cover the sample cell with the instrument cap.



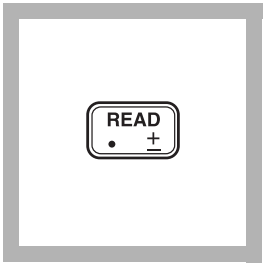
14. Press: **ZERO**

The cursor will move to the right, then the display will show:

0. mg/L TKN



15. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



16. Press: **READ**

The cursor will move to the right, then the result in mg/L total Kjeldahl nitrogen will be displayed.

Note: *Standard Adjust may be performed using a prepared ammonia standard (see Standard Adjust in Section 1).*

$$\text{ppm TKN} = \frac{75 \times A}{B \times C}$$

17. Use the formula shown to calculate the final TKN value.

Where:

A = mg/L displayed

B = g (or mL of water) sample taken for digest

C = mL analysis volume of digested sample (step 5).

Note: *For water samples ppm TKN = mg/L TKN.*

Note: *For maximum accuracy, the reagent blank value may be determined by repeating procedure using reagents only. Subtract the reagent blank value from the reading on the display.*

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Table 1 Analysis Volumes Based on Concentration

AQUEOUS SAMPLES (Solutions of suspensions in water- less than 1% solids)	
Expected Nitrogen Concentration (mg/L)	**Analysis Volume (mL)**
0.5-28	10.00
2-112	5.00
11-560	2.00
45-2250	1.00
425-22500	0.50
DRY SAMPLES	
Expected Nitrogen Concentration (mg/L)	**Analysis Volume (mL)**
42-2200	10.0
106-5600	5.00
350-18000	2.00
1000-56000	1.00
4200-220000	0.50
OILS AND FATS	
Expected Nitrogen Concentration (mg/L)	**Analysis Volume (mL)**
85-4500	10.0
210-11000	5.00
2100-11000	1.00

Sampling and Storage

Collect samples in a cleaned glass or plastic container. Adjust the pH to 2 or less with sulfuric acid (about 2 mL per liter) and cool to 4 °C. Preserved samples can be stored up to 28 days.

Accuracy Check

Kjeldahl Nitrogen Standard Method

This procedure checks digestion efficiency and indicates that amount of bound nitrogen that is freed during digestion. The methods and standards available to check digestion technique are found in the Accuracy Check section following the procedures in the Digesdahl Digestion Apparatus Instruction Manual. Using the digested Kjeldahl standard, perform the above TKN analysis on the colorimeter. The TKN value should come within about ±3% of the value of the prepared Kjeldahl standard.

Standard Solution Method (to check calibration accuracy only)

Add one drop of TKN Indicator to each of two 25-mL graduated mixing cylinders. Fill one cylinder to the 20-mL mark with deionized water. Fill the other cylinder to the 20-mL mark with a 1.0 mg/L Ammonia Nitrogen Solution. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix. Add 3 drops of Polyvinyl Alcohol Dispersing agent to each cylinder. Perform the TKN procedure as described in Steps 9 to 16. This display should show 26-27 mg/L TKN.

User Calibration

For most accurate results, use a user-calibrated program. The Standard Adjust feature should not be used with a user-entered calibration; it will hinder performance.

A one-time setup of a program for TKN is recommended for each new lot of reagents. A new calibration may be performed for each lot of Nessler Reagent by following these instructions:

Standard Preparation

Use the following standards to make a calibration curve. See *Preparing a User-Entered Calibration Curve* on page *49*, for more information and instructions. Prepare standards representing concentrations of 20, 60, 80, 100, 140 and 160 mg/L NH_3-N as follows:

a) Using volumetric pipets, transfer 5.0, 15.0, 20.0, 25.0, 35.0, and 40.0 mL of 100 mg/L NH_3-N standard solution into six separate 100-mL volumetric flasks. Dilute to volume with deionized water, stopper, and invert to mix.

b) Begin at step 4 of the procedure using a 3-mL aliquot for the sample volume. Also prepare a blank solution by substituting a 3 mL aliquot of deionized water for sample in Step 4.

Note: *Standard solutions are prepared as if a 25-mL volume was used for the digestion. Actual concentrations prepared in Step 1 are 5, 15, 20, 25, 35, and*
40 mg/L NH_3-N. These represent original concentrations of 20, 60, 80, 100, 140, and 160 mg/L NH_3-N, based on the 25 to 100 mL dilution in the digestion.

User Entered Calibration Settings For TKN
Program # = 101 to 105
Wavelength = 420 nm
Resolution = 0 mg/L

Method Performance

Precision

In a single laboratory using a standard solution of 64 mg/L TKN and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±1.0 mg/L TKN.

Estimated Detection Limit

The estimated detection limit for program 65 is 2 mg/L TKN. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

"Total Kjeldahl Nitrogen" (also called crude protein) refers to the combination of ammonia and organic nitrogen. Organically-bound in the trinegative state, it is converted into ammonium salts by the action of sulfuric acid and hydrogen peroxide. The ammonia is then analyzed by a modified nessler method test. The Mineral Stabilizer complexes calcium and magnesium. The Polyvinyl Alcohol Dispersing Agent aids the color formation in the reaction of Nessler Reagent with ammonium ions. A yellow color forms, proportional to the ammonia concentration.

Pollution Prevention And Waste Management

Nessler reagent contains mercuric iodide. Both the sample and blank will contain mercury (D009) at concentrations regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See Section *3* for more information on proper disposal of these materials.

REQUIRED REAGENTS

Total Kjeldahl Nitrogen Reagent Set 24953-00
Includes: (1) 21196-49, (1) 23766-26, (1) 21194-49, (1) 23765-26, (1) 282-32H, (1) 23144-26, (1) 979-49, (1) 22519-26

Description	Quantity Required Per Test	Unit	Cat. No.
Hydrogen Peroxide, 50%	20 mL	490 mL	21196-49
Mineral Stabilizer	6 drops	50 mL SCDB	23766-26
Nesslers Reagent	2 mL	500 mL	21194-49
Polyvinyl Alcohol Dispersing Agent	6 drops	50 mL SCDB	23765-26
Potassium Hydroxide Standard Solution, 8.0 N	varies	100 mL MDB	282-32H
Potassium Hydroxide Standard Solution, 1.0 N	varies	50 mL SCDB	23144-26
Sulfuric Acid, ACS	6 mL	500 mL	979-49
TKN Indicator Solution	2 drops	50 mL SCDB	22519-26
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Boiling Chips, silicon carbide	2-3	500 g	20557-34
Cylinder, graduated, mixing, tall-form, 25 mL	2	each	20886-40
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	2	50/pkg	21856-96
Safety Shield, for Digesdahl	1	each	50040-00
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

Select one based on available voltage:

Digesdahl Digestion Apparatus, 115 V	1	each	23130-20
Digesdahl Digestion Apparatus, 230 V	1	each	23130-21

OPTIONAL REAGENTS

Ammonia Nitrogen Standard Solution, 1 mg/L NH_3-N		500 mL	1891-49
Ammonia Nitrogen Standard Solution, Voluette Ampule, 150 mg/L NH_3-N, 10 mL		16/pkg	21284-10
Ammonia Nitrogen Standard Solution, 100 mg/L NH_3-N		500 mL	24065-49
Nitrogen Standard, Primary		3/set	22778-00

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Balance, AccuLab Pocket Pro 250B	each	27969-00
Bottle, glass dispenser, 118 mL	each	591-00
Bottle, plastic wash, 1000 mL	each	620-16
Cylinder, graduated, 50 mL	each	508-41
Flask, volumetric, 100 mL, Class A	each	14574-42
Mini Grinder, 120 V	each	20991-00
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 0.50 mL	each	14515-34
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Pipet, volumetric, Class A, 15.00 mL	each	14515-39
Pipet, volumetric, Class A, 20.00 mL	each	14515-20
Pipet, volumetric, Class A, 25.00 mL	each	14515-40
Safety Glasses	each	18421-00

For Technical Assistance, Price and Ordering

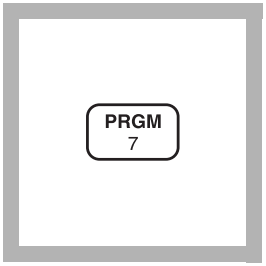
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITROGEN, AMMONIA, Low Range, Test 'N Tube (0 to 2.50 mg/L NH_3-N)

Salicylate Method*

For water, wastewater, and seawater

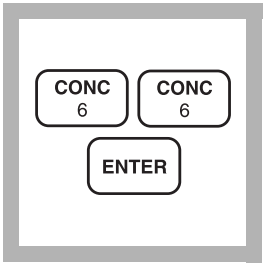


1. Enter the stored program number for low range nitrogen, ammonia Test 'N Tube.

Press: **PRGM**

The display will show:

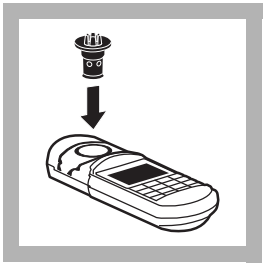
PRGM ?



2. Press: **66 ENTER**

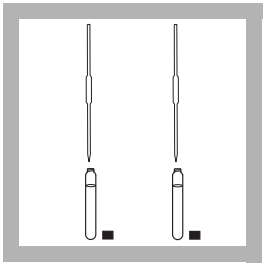
The display will show **mg/L, NH3-N** and the **ZERO** icon**.**

Note: *For alternate forms (NH_3), press the* **CONC** *key.*



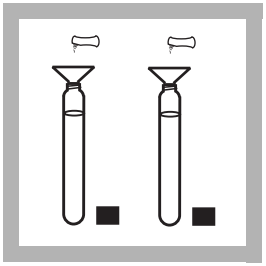
3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

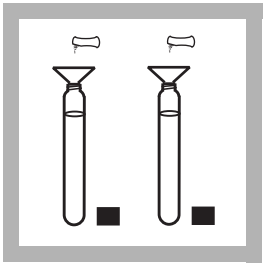


4. Remove the caps from 2 AmVer Diluent Reagent vials. Add 2 mL of sample to one vial (the sample). Add 2 mL of deionized water to the other vial (the blank).

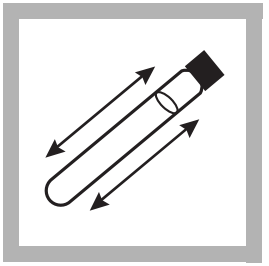
Note: *Adjust the pH of stored samples before analysis. See Interferences on page 365.*



5. Using a funnel, add the contents of one Ammonia Salicylate Reagent Powder Pillow for 5 mL sample to each vial.

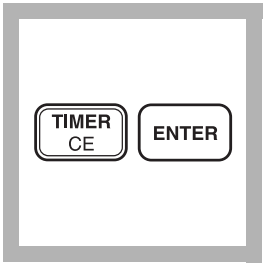


6. Using a funnel, add the contents of one Ammonia Cyanurate Reagent Powder Pillow for 5 mL sample to each vial.



7. Cap the vials tightly and shake thoroughly to dissolve the powder.

Note: *A green color will develop if ammonia is present.*



8. Press:

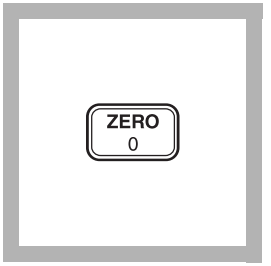
TIMER ENTER

A 20-minute reaction period will begin.

* Adapted from *Clin. Chim. Acta*, 14 403 (1966).

9. Wipe the outside of the vials with a towel. After the timer beeps, place the blank into the adapter. Tightly cover the vial with the instrument cap.

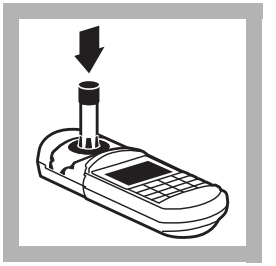
Note: *Wipe with a damp cloth followed by a dry one to remove fingerprints and other marks.*



10. Press: **ZERO**

The cursor will move to the right, then the display will show:

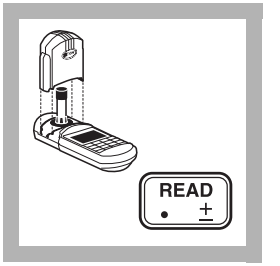
0.00 mg/L NH3-N



11. Place the prepared sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Tightly cover the sample cell with the instrument cap.

Press: **READ**

The cursor will move to the right, then the result in mg/L ammonia nitrogen will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust (Adjusting the Standard Curve) on page 47).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1 N sodium thiosulfate for each 0.3 mg/L Cl_2 in a one liter sample. Preserve the sample by reducing the pH to 2 or less with hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize with 5.0 N sodium hydroxide. Correct the test result for volume additions. See *Correcting for Volume Additions on page 22* for more information.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Nitrogen, Ammonia Ampule Standard Solution, 50 mg/L NH_3-N.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25 mL samples. Mix thoroughly.

c) Analyze each sample as described above. The nitrogen concentration should increase 0.20 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions, Section 1*, for more information.

Standard Solution Method

To check accuracy, use a 1.0 mg/L Nitrogen, Ammonia Standard Solution listed under Optional Reagents. Or, dilute 1 mL of solution from a
50 mg/L Ampule Standard for Nitrogen, Ammonia to 50 mL with deionized water using a 50-mL volumetric flask.

Method Performance

Precision

In a single laboratory, using a standard solution of 1.0 mg/L ammonia nitrogen and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of $\pm$0.02 mg/L NH_3-N.

Estimated Detection Limit

The estimated detection limit for program 66 is 0.08 mg/L NH_3-N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Level and Treatment
Calcium	2500 mg/L as $CaCO_3$
Iron	**1.** Determine the amount of iron present in the sample following one of the total iron procedures. **2.** Add the same iron concentration to the deionized water in step 4. The interference will then be successfully blanked out.
Magnesium	5000 mg/L as $CaCO_3$
Nitrite	30 mg/L as NO_2^--N
Nitrate	250 mg/L as NO_3^--N
Orthophosphate	250 mg/L as PO_4^{3-}-P
pH	Acidic or basic samples should be adjusted to about pH 7. Use 1 N Sodium Hydroxide Standard Solution for acidic samples and 1 N Hydrochloric Acid Standard Solution for basic samples.
Sulfate	300 mg/L as SO_4^{2-}

Interfering Substance	Interference Level and Treatment
Sulfide	**1.** Measure about 350 mL of sample in a 500 mL erlenmeyer flask. **2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix. **3.** Filter the sample through a folded filter paper. **4.** Use the filtered solution in step 4.
Other	Less common interferences such as **hydrazine** and **glycine** will cause intensified colors in the prepared sample. **Turbidity** and **color** will give erroneous high values. Samples with severe interferences require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set. See Optional Apparatus at the end of this procedure.

Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

Pollution Prevention And Waste Management

The ammonia salicylate reagent contains sodium nitroferricyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Collect cyanide solutions for disposal as reactive (D001) waste. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. See *Section 3* for further information in proper disposal of these materials.

REQUIRED REAGENTS

	Cat. No.
AmVer Reagent Set for Nitrogen, Ammonia, Low Range TNT (25 tests) Includes: (1) 23952-66, (1) 23954-66, (1) 272-42, *(50) AmVer Low Range Vials	26045-45

Description	Quantity Required Per Test	Unit	Cat. No.
AmVer Diluent Reagent, Low Range Test 'N Tube	2 vials	50/pkg	*
Salicylate Reagent Powder Pillows, 5 mL sample	2 pillows	50/pkg	23952-66
Cyanurate Reagent Powder Pillows, 5 mL sample	2 pillows	50/pkg	23954-66

* Not available separately.

REQUIRED APPARATUS

Vial Adapter, COD	1	each	48464-00
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette, 0-10 mL	1	each	19700-10
Pipet Tips for 19700-10	2	50/pkg	21997-96
Funnel, micro (for reagent addition)	1	each	25843-35

OPTIONAL REAGENTS

Nitrogen, Ammonia Standard Solution, 1.0 mg/L NH_3-N		500 mL	1891-49
Nitrogen, Ammonia Standard Solution, 10 mL Voluette ampules, 50 mg/L NH_3-N		16/pkg	14791-10
Nitrogen, Ammonia Standard Solution, 2 mL PourRite ampules, 50 mg/L NH_3-N		20/pkg	14791-20
Hydrochloric Acid, ACS		500 mL	134-49
Sodium Hydroxide Standard Solution, 5.0 N		50 mL SCDB	2450-26
Sodium Hydroxide, 1.000 N		100 mL MDB	1045-32
Sodium Thiosulfate Standard Solution, 0.1 N		100 mL MDB	323-32
Sulfide Inhibitor Reagent Powder Pillows		100/pkg	2418-99
Sulfuric Acid, 1.00 N		100 mL MDB	1270-32
Wastewater Effluent Standard, Inorganics (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28332-49
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit		each	21968-00
Cylinder, graduated, mixing, 25 mL, Class A		each	508-40
Distillation Apparatus Set		each	22653-00
Heater and Support Apparatus (for distillation), 115 Vac		each	22744-00
Heater and Support Apparatus (for distillation), 230 Vac		each	22744-02
Filter Paper, folded		100/box	1894-57
Flask, Erlenmeyer, 500 mL		each	505-49
Flask, volumetric, 50 mL, Class A		each	14547-41
Funnel, analytical (for filtering)		each	1083-68
Jack, laboratory (use with distillation apparatus)		each	22743-00
pH Indicator Paper, 1 to 11 pH		5 rolls/pkg	391-33
Ampule Breaker Kit, PourRite		each	24846-00
Thermometer, –20 to 110 °C, non-mercury		each	26357-02
Thermometer, –10 to 260 °C, non-mercury		each	26357-01

For Technical Assistance, Price and Ordering

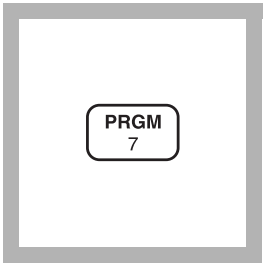
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITROGEN, AMMONIA, High Range, Test 'N Tube

(0 to 50 mg/L NH_3-N) **For water, wastewater, and seawater**

Salicylate Method*

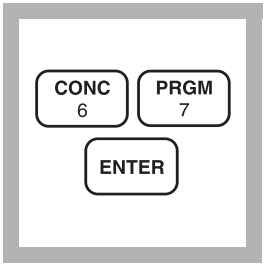


1. Enter the stored program number for nitrogen, ammonia, high range Test 'N Tube (NH_3-N) method.

Press: **PRGM**

The display will show:

PRGM ?

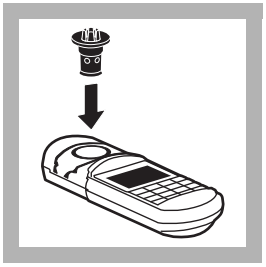


2. Press: **67 ENTER**

The display will show **mg/L, NH3-N** and the **ZERO** icon**.**

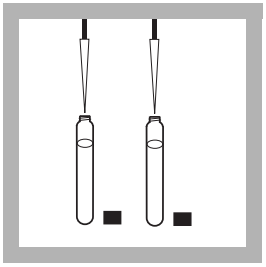
Note: *For alternate forms (NH_3), press the* **CONC** *key.*

Note: *For proof of accuracy, use a 10-mg/L nitrogen, ammonia standard in place of the sample.*

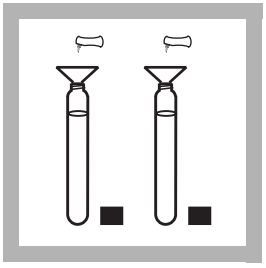


3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

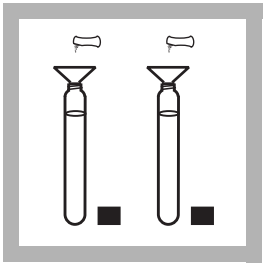
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



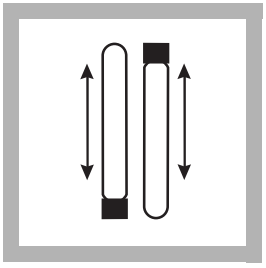
4. Remove the caps from 2 AmVer Diluent Reagent High Range Vials. Add 0.1 mL of sample to one vial (the sample). Add 0.1 mL of deionized water to the other (the blank).



5. Add the contents of 1 Ammonia Salicylate Reagent Powder Pillow for 5 mL Sample to each vial.

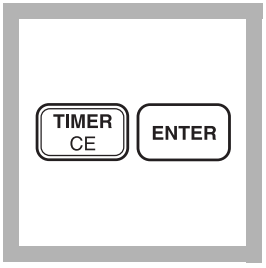


6. Add the contents of 1 Ammonia Cyanurate Reagent Powder Pillow for 5 mL Sample to each vial.



7. Cap the vials tightly and shake thoroughly to dissolve the powder.

Note: *A green color will develop if ammonia is present.*

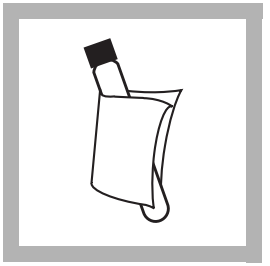


8. Press:

TIMER ENTER

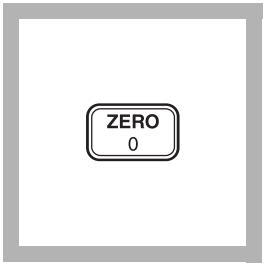
A 20-minute reaction period will begin.

* Adapted from *Clin. Chim. Acta*, **14** 403 (1966).



9. Clean the outside of the vial with a towel. After the timer beeps, place the blank into the vial adapter. Tightly cover the vial with the instrument cap.

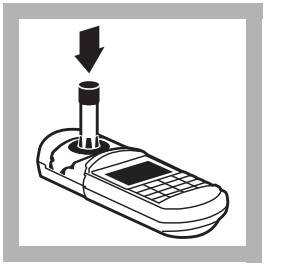
Note: *Wipe with a damp cloth and follow with a dry one to remove fingerprints and other marks.*



10. Press: **ZERO**

The cursor will move to the right, then the display will show:

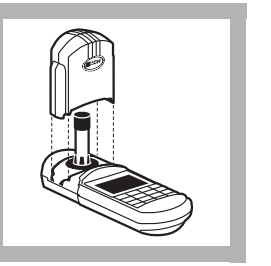
0 mg/L NH3-N



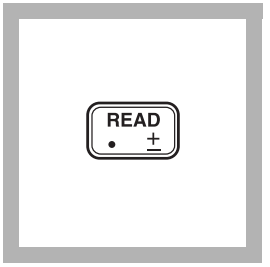
11. Place the prepared sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Tightly cover the vial with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L NH_3-N will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis. If chlorine is known to be present, add one drop of 0.1 N sodium thiosulfate for each 0.3 mg/L Cl_2 in a one liter sample. Preserve the sample by reducing the pH to 2 or less with hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Before analysis, warm samples to room temperature and neutralize with 5.0 N sodium hydroxide. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method

a) Snap the top off an Ammonia PourRite Ampule Standard, 150 mg/L NH_3-N.

b) Use the TenSette Pipet to add 0.2, 0.4 and 0.6 mL of standard to three 25-mL samples. Swirl to mix.

c) Analyze each sample as described above. The ammonia concentration should increase approximately 1.2 mg/L NH_3-N for each 0.2 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To check accuracy, use a 10 or 50 mg/L Nitrogen, Ammonia Standard Solution or use a Nitrogen, Ammonia Voluette Ampule Standard, 50 mg/L.

Method Performance

Precision

In a single laboratory, using a standard solution of 50 mg/L ammonia nitrogen (NH_3-N) and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of $\pm$5 mg/L NH_3-N.

Estimated Detection Limit

The estimated detection limit for program 67 is 1 mg/L NH_3-N. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following ions may interfere when present in concentrations exceeding those listed below.

In some lab environments, airborne cross contamination of the blank is possible. Complete preparation of the blank before opening or handling any samples or standards to avoid transfer of ammonia. If sample or standard containers have already been open, move to a separate area of the lab to prepare the blank.

Substance	Concentration and Suggested Treatments
Acidic or basic samples	Adjust to approximately pH 7. Use 1 N Sodium Hydroxide Standard Solution for acidic samples and 1 N Hydrochloric Acid Standard Solution for basic samples.
Calcium	50,000 mg/L as $CaCO_3$
Glycine, hydrazine	Will cause intensified colors in the prepared sample.
Magnesium	300,000 mg/L as $CaCO_3$
Iron	Eliminate iron interference as follows: **1.** Determine the amount of iron present in the sample using one of the total iron procedures. **2.** Add the same iron concentration to the deionized water in step 4. **3.** The interference will then be successfully blanked out.
Nitrite	600 mg/L as NO_2^--N
Nitrate	5,000 mg/L as NO_3^--N
Orthophosphate	5,000 mg/L as PO_4^{3-}-P
Sulfate	6,000 mg/L as SO_4^{2-}
Sulfide	Sulfide will intensify the color. Eliminate sulfide interference as follows: **1.** Measure about 350 mL of sample in a 500 mL Erlenmeyer flask. **2.** Add the contents of one Sulfide Inhibitor Reagent Powder Pillow. Swirl to mix. **3.** Filter the sample through folded filter paper. Use the filtered solution in step 4.
Turbidity and color	Give erroneous high values. Samples with severe interferences require distillation. Hach recommends the distillation procedure using the Hach General Purpose Distillation Set.

Summary of Method

Ammonia compounds combine with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a green-colored solution.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheets* for information specific to the reagents used. For additional information, refer to *Section 3*.

Pollution Prevention And Waste Management

The ammonia salicylate reagent contains sodium nitroferricyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Collect cyanide solutions for disposal as reactive (D001) waste. Be sure cyanide solutions are stored in a caustic solution with pH >11 to prevent release of hydrogen cyanide gas. See *Section 3* for further information in proper disposal of these materials.

REQUIRED REAGENTS

AmVer™ Reagent Set for Nitrogen, Ammonia, High Range, TNT (25 tests) 26069-45
Includes: (1) 23952-66, (1) 23954-66, (1) 272-42, *(50) AmVer HR Vials

Description	Quantity Required Per Test	Unit	Cat. No.
AmVer™ HR Reagent Test 'N Tube™ Vials	2 vials	50/pkg	*
Ammonia Salicylate Reagent Powder Pillows	2 pillows	50/pkg	23952-66
Ammonia Cyanurate Reagent Powder Pillows	2 pillows	50/pkg	23954-66

REQUIRED APPARATUS

COD/TNT Adapter	1	each	48464-00
Pipet, TenSette®, 0-1 mL	1	each	19700-01
Pipet Tips for 19700-01	varies	50/pkg	21856-96
Test Tube Rack	1-3	each	18641-00
Funnel, micro (for reagent addition)	1	each	25843-35

* Not available separately.

OPTIONAL REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Nitrogen, Ammonia Standard Solution, 50 mg/L NH_3-N		500 mL	14791-50
Nitrogen, Ammonia Standard Solution, 10 mg/L NH_3-N		500 mL	153-49
Ammonia Standard Solution, PourRite™ ampules, 150 mg/L NH_3-N, 2 mL		20/pkg	21284-20
Hydrochloric Acid, ACS		500 mL	134-49
Sodium Hydroxide Standard Solution, 5.0 N		50 mL	2450-26
Sodium Hydroxide Standard Solution, 1.0 N		100 mL	1045-32
Sodium Thiosulfate Standard Solution, 0.1 N		100 mL	323-32
Sulfide Inhibitor Powder Pillows		100/pkg	2418-99
Sulfuric Acid, 1.00 N		100 mL MDB	1270-32
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3, PO_4, COD, SO_4, TOC)		500 mL	28331-49
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

Cylinder, 25 mL, graduated, mixing		each	20886-40
Distillation Apparatus Set, general purpose		each	22653-00
Heater and Support Apparatus (for distillation), 115 VAC		each	22744-00
Heater and Support Apparatus (for distillation), 230 VAC		each	22744-02
Filter Paper, folded		100/pkg	1894-57
Flask, Erlenmeyer, 500 mL		each	505-49
Funnel, analytical (for filtering)		each	1083-68
Jack, laboratory (use with distillation apparatus)		each	22743-00
pH Indicator Paper, 1 to 11 pH		5 rolls/pkg	391-33
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite™ Ampule Breaker		each	24846-00
Sample Cell, 10-20-25 mL, w/cap		6/pkg	24019-06

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

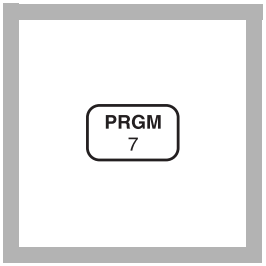
Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITROGEN, Total Inorganic, Test 'N Tube™ (0 to 25.0 mg/L N)

Titanium Trichloride Reduction Method

For water, wastewater, and seawater

Requires Centrifuge

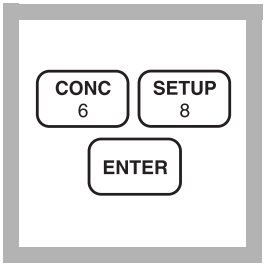


1. Enter the stored program number for Test 'N Tube Total Inorganic Nitrogen.

Press: **PRGM**

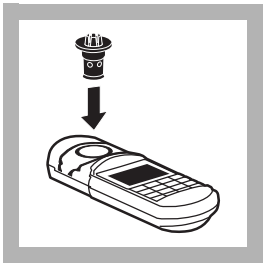
The display will show:

PRGM ?



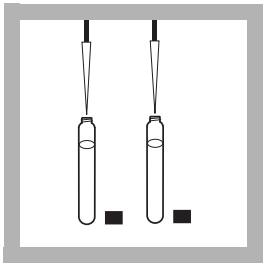
2. Press: **68 ENTER**

The display will show **mg/L, N** and the **ZERO** icon**.**

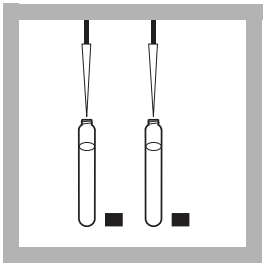


3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert.

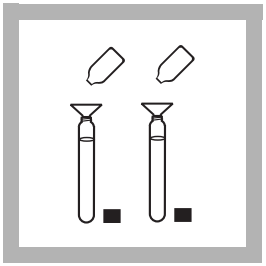
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



4. Pipet 1 mL of Total Inorganic Nitrogen Pretreatment Base Concentrate into each of 2 Total Inorganic Nitrogen Pretreatment Diluent Vials.



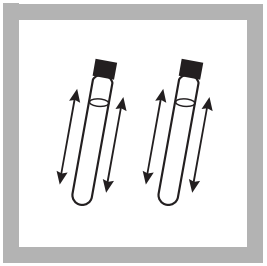
5. Pipet 1 mL of sample into 1 TIN Diluent Vial (the sample). Pipet 1 mL of deionized water into the other vial (the blank). Cap the vials and shake for 30 seconds to mix.



6. Snap the necks off two Total Inorganic Nitrogen Reductant ampules and pour the contents of one into the TIN Diluent Vial containing sample. Repeat for the second vial, the blank.

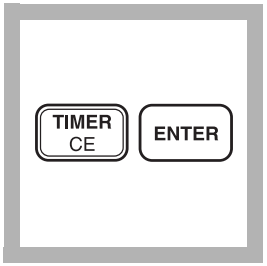
Note: *For safety, wear gloves while breaking the ampules.*

Note: *A black precipitate will form immediately.*



7. Cap the vials. Shake gently for 30 seconds to mix the reagents. Allow the vials to sit for at least one minute.

Note: *The precipitate should remain black after shaking. Excessive shaking will cause a white precipitate and low results.*

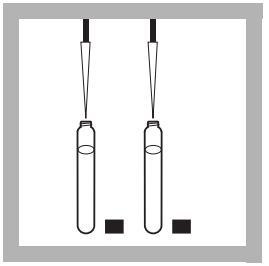


8. Centrifuge the vials for 3 minutes or until the solids settle to the bottom of the vial.

Press: **TIMER ENTER**

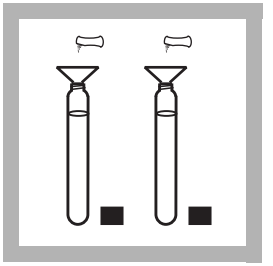
immediately after starting the centrifuge.

Note: *The precipitate will settle without using a centrifuge, but it may take up to 30 minutes.*

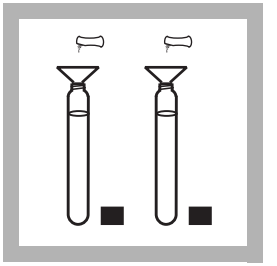


9. Remove the caps from 2 AmVer Diluent Reagent Test 'N Tubes for Low Range Ammonia Nitrogen. Using a pipet, add 2 mL of centrifuged sample into 1 vial. Add 2 mL of centrifuged blank to the other vial. Label the vials appropriately.

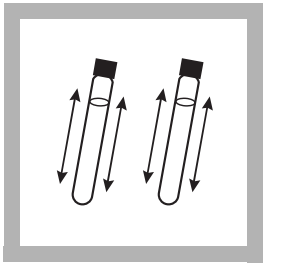
Note: *Pipet carefully to avoid disturbing the sediment.*



10. Using a funnel, add the contents of one Ammonia Salicylate Reagent Powder Pillow to each vial.

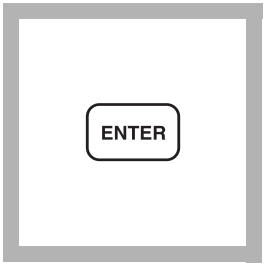


11. Using a funnel, add the contents of one Ammonia Cyanurate Reagent Powder Pillow to each vial.



12. Cap the vials tightly and shake thoroughly to dissolve the powder.

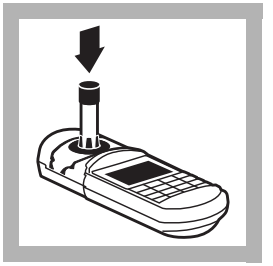
Note: *A green color will develop if inorganic nitrogen is present.*



13. The display will show: **15:00 TIMER 2**

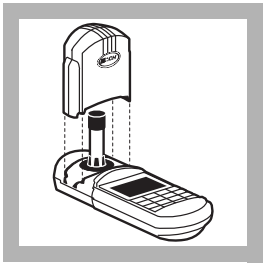
Press: **ENTER**

A 15-minute reaction period will begin.

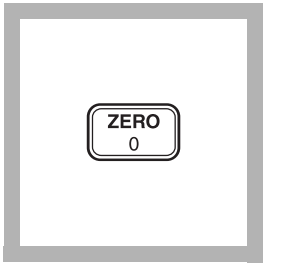


14. After the timer beeps, clean the outside of the vials with a towel. Place the blank in the adapter. Push straight down on the top of the vial until it seats solidly into the adapter. Do not move the vial from side to side as this can cause errors.

Note: *Wipe with a damp cloth and follow with a dry one to remove fingerprints and other marks.*



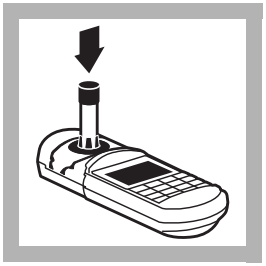
15. Tightly cover the sample cell with the instrument cap.



16. Press: **ZERO**

The cursor will move to the right, then the display will show:

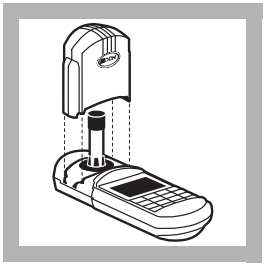
0.0 mg/L N



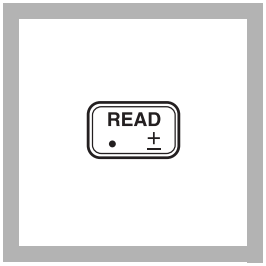
17. Place the prepared sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



18. Tightly cover the sample cell with the instrument cap.



19. Press: **READ**

The cursor will move to the right, then the result in mg/L total inorganic nitrogen will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling And Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

If chlorine is known to be present, add 1 drop of 0.1 N sodium thiosulfate for each 0.3 mg/L Cl_2 in a 1 liter sample.

Preserve the sample by reducing the pH to 2 or less with concentrated hydrochloric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N Sodium Hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1*.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a fresh High Range Nitrate Nitrogen PourRite Ampule Standard, 500 mg/L NO_3^--N.

c) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to 3 25-mL mixing cylinders. Mix thoroughly.

d) Analyze each sample as described in the procedure; use a 1-mL aliquot of the prepared sample in Step 5. The nitrogen concentration should increase about 1.8 to 1.9 mg/L for each 0.1 mL of standard added.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To check accuracy, use a 10.0 mg/L Nitrate Nitrogen Standard Solution listed under Optional Reagents. Alternatively, a 20.0 mg/L nitrate nitrogen standard can be prepared by diluting 2 mL of solution from a PourRite Ampule Standard for High Range Nitrate Nitrogen, 500 mg/L NO_3^--N, to 50 mL with deionized water. Substitute this standard for the sample and perform the test as described. The recovery of the standards should be about 90-95%.

Method Performance

Precision/Accuracy

The total inorganic nitrogen test provides an estimate of the total nitrite, nitrate, and ammonia nitrogen load in water or wastewater samples. This test is most applicable for monitoring an industrial process stream or a wastewater treatment stream where it is important to track the inorganic nitrogen load as it passes through the treatment process. The test exhibits different recoveries of each of the three nitrogen species, as summarized below. This test is not recommended for quantifying only one of the three species. In that case, use a specific procedure for each particular analyte.

Ammonia Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NH_3^-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 21.3 mg/L with a standard deviation of
± 0.77 mg/L N (replicate number = 7 per reagent lot).

Nitrate Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NO_3^-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 18.9 mg/L with a standard deviation of
± 0.55 mg/L N (replicate number = 7 per reagent lot).

Nitrite Nitrogen

In a single laboratory, using a standard solution of 20.0 mg/L NO_2^-N and 2 representative lots of reagent with the instrument, a single operator obtained a mean recovery of 14.6 mg/L with a standard deviation of
± 0.77 mg/L N (replicate number = 7 per reagent lot).

Estimated Detection Limit
The estimated detection limit for program 68 is 0.7 mg/L N. For more information on the estimated detection limit, *see Section 1*.

Interferences

The following ions may interfere when present in concentrations exceeding those listed below:

Species	Level	Effect
Calcium	1000 mg/L as $CaCO_3$	Positive
Manganese (IV)	3 mg/L	Negative
Magnesium	1000 mg/L as $CaCO_3$	Positive
Sulfide	3 mg/L	Negative
Sulfate	250 mg/L	Negative

The following do not interfere below the levels listed:

Species	Level
Al^{3+}	8 mg/L
Ba^{2+}	40 mg/L
Cu^{2+}	40 mg/L
Fe^{3+}	8 mg/L
Zn^{2+}	80 mg/L
F^-	40 mg/L
PO_4^{3-}-P	8 mg/L
SiO_2	80 mg/L
EDTA	80 mg/L

Summary of Method

Titanium (III) ions reduce nitrate and nitrite to ammonia in a basic environment. After centrifugation to remove solids, the ammonia is combined with chlorine to form monochloramine. Monochloramine reacts with salicylate to form 5-aminosalicylate. The 5-aminosalicylate is oxidized in the presence of a sodium nitroprusside catalyst to form a blue-colored compound. The blue color is masked by the yellow color from the excess reagent present to give a final green-colored solution.

REQUIRED REAGENTS

Total Inorganic Nitrogen Pretreatment Reagent Set ($TiCl_3$ Reduction) (25 tests)..... 26049-45
Includes: (1) 26051-50, (1) 2040-59, *(50) TIN Pretreatment Diluent Vials

AmVer™ Reagent Set for Nitrogen, Ammonia, Low Range (25 tests)..... 26045-45
Includes: (1) 23952-66, (1) 23954-66 , (1) 272-42, *(50) AmVer™ Diluent LR Vials

Description	Quantity Required Per Test	Unit	Cat. No.
Total Inorganic Nitrogen Pretreatment Diluent Vials	2 vials	50/pkg	*
Total Inorganic Nitrogen Reductant Ampules	2 ampules	50/pkg	26051-50
Total Inorganic Nitrogen Pretreatment Base Concentrate	2 mL	50 mL	2040-59
AmVer™ Diluent Reagent, Low Range Vials	2 vials	50/pkg	*
Ammonia Salicylate Reagent Powder Pillows for 5-mL sample	2 pillows	50/pkg	23952-66
Ammonia Cyanurate Reagent Powder Pillows for 5-mL sample	2 pillows	50/pkg	23954-66

REQUIRED APPARATUS

Centrifuge, 115V	1	each	26765-00
Centrifuge, 230V	1	each	26765-02
COD/TNT Vial Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette®, 0.1 to 1.0	1	each	19700-01
Pipet Tips for 19700-01	2	50/pkg	21856-96
Test Tube Rack	1	each	18641-00

OPTIONAL REAGENTS

Hydrochloric Acid, ACS	500 mL	134-49
Nitrate Nitrogen Standard Solution, 10 mg/L NO_3^--N	500 mL	307-49
Nitrate Nitrogen Standard Solution, PourRite Ampules, 500 mg/L NO_3^--N, 2 mL	20/pkg	14260-20
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB	2450-26
Sodium Thiosulfate Standard Solution, 0.1 N	100 mL MDB	323-32
Wastewater Effluent Standard, Inorganics (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Water, deionized	4 L	272-56

* These items are not sold separately. Please order the complete set (cat. no. 26049-45 or 26045-45).

OPTIONAL APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Cylinder, graduated, mixing, 25 mL		each	20886-40
Flask, volumetric, Class A, 50.0 mL		each	14574-41
pH Indicator Paper, 1 to 11 pH		5 rolls/pkg	391-33
Pipet, volumetric, Class A, 2.0 mL		each	14515-36
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite Ampule Breaker		each	24846-00

For Technical Assistance, Price and Ordering

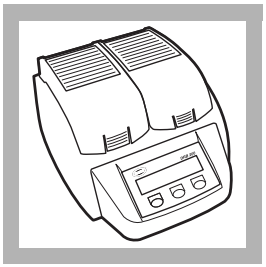
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

NITROGEN, TOTAL, Test 'N Tube (0.0 to 25.0 mg/L N)

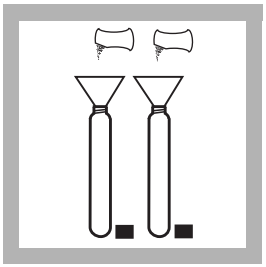
TNT Persulfate Digestion Method

For water and wastewater



1. Turn on the DRB 200 Reactor. Heat to 103-106 °C (optimum temperature is 105 °C).

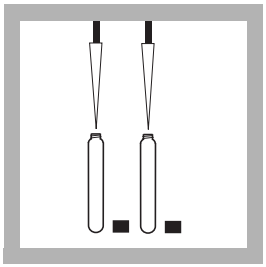
Note: *For proof of accuracy, run a 20 mg/L NH_3-N standard through digestion and analysis.*



2. Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to each of two Total Nitrogen Hydroxide Reagent vials.

Note: *Wipe off any reagent that may get on the lid or the tube threads.*

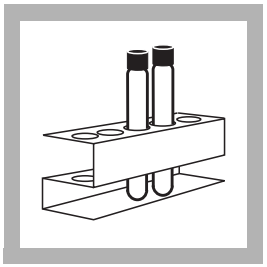
Note: *One reagent blank is sufficient for each set of samples.*



3. Add 2 mL of sample to one vial. Add 2 mL of organic-free water to another vial (the reagent blank). Cap both vials and shake vigorously (about 30 seconds). Place the vials in the Reactor. Heat for 30 minutes.

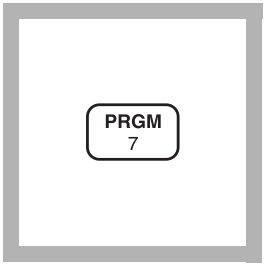
Note: *The reagent may not dissolve completely after shaking.*

Note: *Alternate water must be free of all nitrogen-containing species.*



4. Using finger cots or gloves, remove the hot vials from the reactor and allow to cool to room temperature.

Note: *It is very important to remove the vials from the Reactor after exactly 30 minutes.*

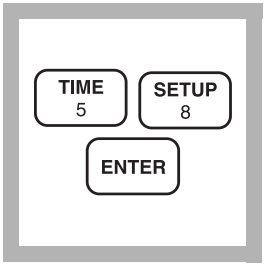


5. Enter the stored program number for Test ‘N Tube Total Nitrogen.

Press: **PRGM**

The display will show:

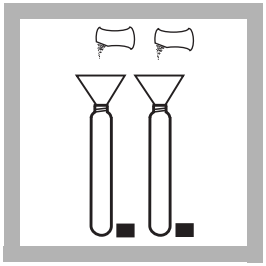
PRGM ?



6. Press: **58 ENTER**

The display will show **mg/L, N** and the **ZERO** icon**.**

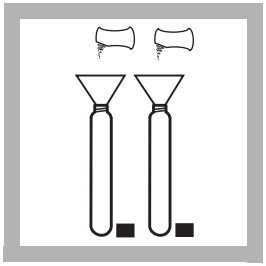
Note: *For alternate forms (NH_3, NO_3), press the* **CONC** *key.*



7. Remove the caps from the digested vials and add the contents of one TN Reagent A Powder Pillow to each vial. Cap the vials and shake for 15 seconds.

Press: **TIMER ENTER** after shaking.

A three-minute reaction period will begin.



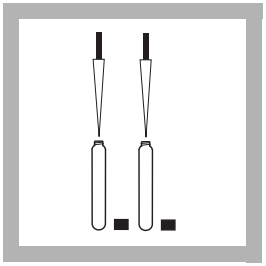
8. After the timer beeps, remove the caps from the vials and add one TN Reagent B Powder Pillow to each vial. Cap the vials and shake for 15 seconds. The display will show:

02:00 Timer 2

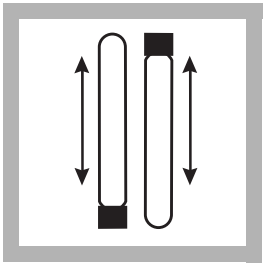
Press **ENTER** after shaking.

A two-minute reaction period will begin.

Note: *The reagent will not completely dissolve. The solution will begin to turn yellow.*

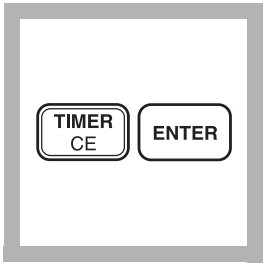


9. After the timer beeps, remove the caps from two TN Reagent C Vials. Add 2 mL of digested, treated sample to one vial. Add 2 mL of the digested, treated reagent blank to the second TN Reagent C Vial.



10. Cap and invert 10 times to mix. Use slow, deliberate inversions for complete recovery. The vials will be warm.

Note: *Follow these instructions for inversion or low results may occur. Hold the vial vertical with the cap up. Invert the vial and wait for all of the solution to flow to the cap end. Pause. Return the vial to the upright position and wait for all of the solution to flow to the vial bottom. This is one inversion (10 inversions = 30 seconds).*

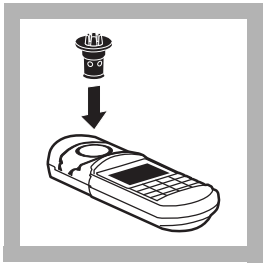


11. The display will show: **05:00 Timer 3**

Press: **ENTER**

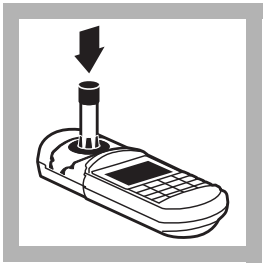
A five-minute reaction period will begin.

Note: *The yellow color will intensify.*



12. During the reaction period, insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

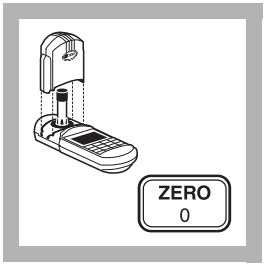
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



13. After the timer beeps, wipe the TN Reagent C vial containing the reagent blank. Place the vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



14. Tightly cover the vial with the instrument cap.

Press: **ZERO**

The cursor will move to the right, then the display will show:

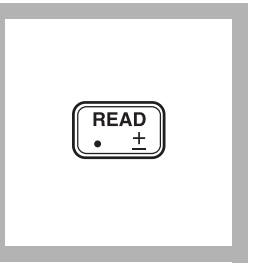
0.0 mg/L N

Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*

Note: *The reagent blank is stable when stored in the dark; see* Blanks For Colorimetric Measurement *following these steps.*

15. Wipe the TN Reagent C vial containing the sample and place it into the adapter. Tightly cover the vial with the instrument cap.

Note: *Multiple samples may be read after zeroing on one reagent blank.*



16. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrogen (N) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Note: *If the display flashes "limit", dilute the sample and repeat the digestion and the colorimetric finish. The digestion must be repeated for accurate results; diluting and repeating the color finish does not yield complete results. Multiply the result by the dilution factor; see Section 1.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated sulfuric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1*.

Accuracy Check

This method generally yields 95-100% recovery on organic nitrogen standards. For proof of accuracy Hach offers a set of three Primary Standards for Kjeldahl Nitrogen.

1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 25 mg/L N standard. Use water that is free of all organic and nitrogen-containing species.

   a) Weigh 0.3379 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   b) Weigh 0.4416 g of Glycine p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   c) Weigh 0.5274 g of Nicotinic p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

2. Analyze each of these solutions using the test procedure above. Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{25} \times 100$$

The percent recovery should be:

Compound	Lowest Expected % Recovery
Ammonia-PTSA	95%
Glycine-PTSA	95%
Nicotinic-PTSA	95%

Hach analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

Standard Solution Method

Substitute 2 mL of a 20 mg/L ammonia nitrogen standard solution for the sample. To prepare a 20-mg/L standard, use a 20-mL Class A pipet to transfer 20 mL of a 100-mg/L Ammonia Nitrogen Standard (see *Optional Reagents*) to a 100-mL Class A volumetric flask. Dilute to the line with organic-free water. A single analyst should obtain less than 5% variation on replicates. Comparison of the user-obtained value with the standard concentration is an indication of test performance for this user.

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off an Ammonia Nitrogen Voluette Ampule Standard Solution, 160 mg/L as NH_3-N.

c) Use the TenSette Pipet to add 0.3 mL, 0.6 mL, and 0.9 mL of standard, respectively, to the three mixing cylinders.

d) Stopper each cylinder and mix thoroughly.

e) Add 2 mL of each prepared solution, respectively, to three TN Hydroxide Reagent Sample Digestion Vials.

f) Analyze each standard addition sample as described in the procedure. The nitrogen concentration should increase 2 mg/L for each 0.3 mL of standard added.

g) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

Blanks for Colorimetric Measurement

The reagent blank may be used up to 7 days for measurements using the same lots of reagents. Store the reagent blank in the dark at room temperature (18-25 °C). If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

Method Performance

Precision

A Hach chemist analyzed two independent nutrient standards. The lowest average percent recovery was 95% with a standard deviation of ±2%.

In a single laboratory, using a standard solution of 15.0 mg/L N and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than ±0.5 mg/L N. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 58 is 2 mg/L N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering substances that resulted in a concentration change of ±10%:

Substance	Level and Effect
Bromide	>60 ppm; positive interference
Chloride	>1000 ppm; positive interference

The substances in the following table have been tested and found **not** to interfere up to the indicated levels (in mg/L):

Substance	Maximum Level Tested (mg/L)
Barium	2.6
Calcium	300
Chromium (3+)	0.5
Iron	2
Lead	6.6 ppb
Magnesium	500
Organic Carbon	150
pH	13 pH units
Phosphorus	100
Silica	150
Silver	0.9
Tin	1.5

Hach chemists tested this chemistry on standard nitrogen solutions prepared from the following compounds and obtained ≥95% recovery:

- Ammonium chloride
- Ammonium sulfate
- Ammonium acetate
- Urea
- Glycine

Ammonium chloride or nicotinic-PTSA spikes in domestic influent, effluent and the ASTM standard specification for substitute wastewater (D 5905-96) also resulted in ≥95% recovery.

Large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum near 420 nm.

REQUIRED REAGENTS

Description	Cat. No.
Test 'N Tube Total Nitrogen Reagent Set (50 vials)	26722-45
Includes:	
TN Reagent C Vials, Acid Solution*, 50/pkg	26721-45
TN Hydroxide Reagent Sample Digestion Vials*, 50/pkg	26717-45

Description	Quantity Required Per Test	Unit	Cat. No.
TN Persulfate Reagent Powder Pillows	2 pillows	100/pkg	26718-49
TN Reagent A, Bisulfite Powder Pillows	2 pillows	100/pkg	26719-49
TN Reagent B, Indicator Powder Pillows	2 pillows	100/pkg	26720-49

REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
COD/TNT Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette, 1.0-10.0 mL	1	each	19700-10
Pipet Tips for 19700-10	1	50/pkg	21997-96
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips for 19700-01	2	50/pkg	21856-96
Test Tube Cooling Rack	1-3	each	18641-00

* Not available separately.

OPTIONAL REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Nitrogen,Ammonia, 100 mg/L NH_3-N	500 mL	24065-49	
Nitrogen, Ammonia, Voluette Ampule, 160 mg/L NH_3–N, 10 mL		16/pkg	21091-10
Sulfuric Acid, ACS		500 mL	979-49
Primary Standards for Kjeldahl Nitrogen		set of 3	22778-00
Ammonium p-Toluenesulfonate		25 g	22779-24
Glycine p-Toluenesulfonate		25 g	22780-24
Nicotinic Acid p-Toluenesulfonate		25 g	22781-24
Sodium Hydroxide Standard Solution, 5.0 N		50 mL MDB	2450-26
Wastewater Effluent Standard, Inorganics (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28332-49
Water, organic-free		500 mL	26415-49

OPTIONAL APPARATUS

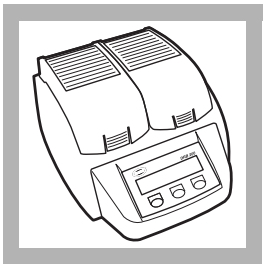
Ampule Breaker Kit	each	21968-00
Balance, analytical, 115 VAC	each	28014-01
Balance, analytical, 230 VAC	each	28014-02
Cots, finger	2/pkg	14647-02
Cylinder, graduated, mixing, 25 mL (3 required)	each	26363-40
Flask, volumetric, Class A, 1000 mL (3 required)	each	14574-53
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, volumetric, Class A, 20 mL	each	14515-20
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33

DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm	LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm	LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm	LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm	LTV082.52.30001

NITROGEN, TOTAL, HR, Test 'N Tube™ (10.0 to 150.0 mg/L N)

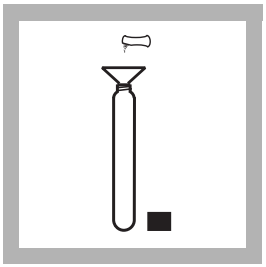
TNT Persulfate Digestion Method

For water and wastewater



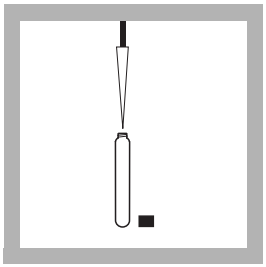
1. Turn on the DRB 200 Reactor. Heat to 103-106 °C (optimum temperature is 105 °C).

Note: *For proof of accuracy, run a 125 mg/L NH_3-N standard through digestion and analysis.*



2. Prepare a reagent blank: Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to one HR Total Nitrogen Hydroxide Digestion Vial.

Note: *Wipe off any reagent that gets on the lid or the tube threads.*



3. Add 0.5 mL of organic-free water to the vial. Cap the vial and shake vigorously for about 30 seconds.

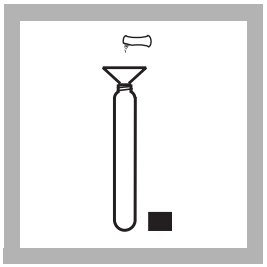
Process this reagent blank exactly the same as the sample, including digestion and color finish. Proceed to *step 6.*

Note: *Alternate water must be free of all nitrogen-containing species.*

Note: *The persulfate reagent may not dissolve completely after shaking.*

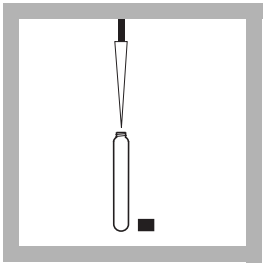
Note: *One reagent blank is sufficient for each set of samples using the same lots of reagents.*

Note: *The reagent blank is stable for as long as seven days when stored in the dark; see* Blanks for Colorimetric Measurement *following this procedure.*



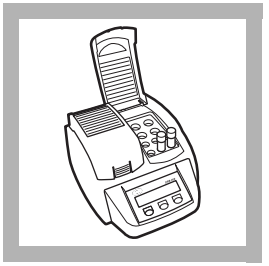
4. Prepare a sample: Using a funnel, add the contents of one Total Nitrogen Persulfate Reagent Powder Pillow to one HR Total Nitrogen Hydroxide Digestion Vial.

Note: *Wipe off any reagent that gets on the lid or the tube threads.*

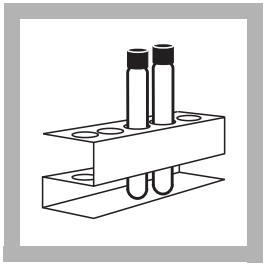


5. Add 0.5 mL of sample to the vial. Cap the vial and shake vigorously for about 30 seconds.

Note: *The persulfate reagent may not dissolve completely after shaking.*

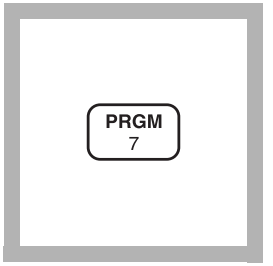


6. Place the vials in the Reactor. Heat for 30 minutes.



7. Using finger cots or gloves, remove the hot vials from the reactor and allow to cool to room temperature.

Note: *It is very important to remove the vials from the Reactor after exactly 30 minutes.*

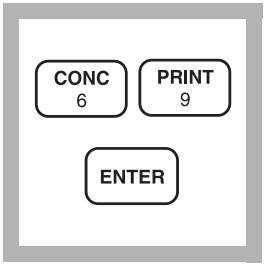


8. Enter the stored program number for Test 'N Tube HR Total Nitrogen.

Press: **PRGM**

The display will show:

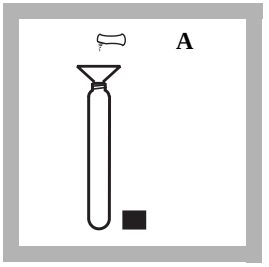
PRGM ?



9. **Press: 69 ENTER**

The display will show **mg/L, N** and the **ZERO** icon**.**

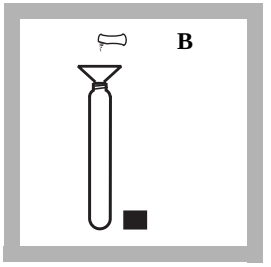
Note: *For alternate forms (NH_3, NO_3), press the* **CONC** *key.*



10. Add the contents of one Total Nitrogen Reagent A Powder Pillow to the vial containing the digested blank or sample. Cap the vial and shake for 15 seconds.

Press: **TIMER ENTER** after shaking.

A three-minute reaction period will begin.



11. After the timer beeps, add one Total Nitrogen Reagent B Powder Pillow to the vial. Cap the vial and shake for 15 seconds.

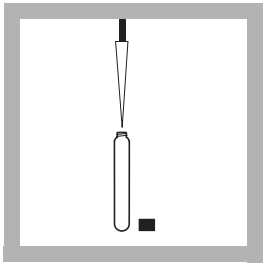
The display will show:

02:00 Timer 2

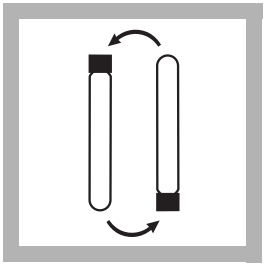
Press **ENTER** after shaking.

A two-minute reaction period will begin.

Note: *The reagent will not completely dissolve. The solution will begin to turn yellow.*

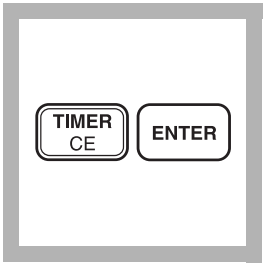


12. After the timer beeps, remove the cap from one Total Nitrogen Reagent C Vial. Add 2 mL of digested, treated sample (or reagent blank) to the vial. The vial will be warm.



13. Cap and invert slowly 10 times to mix. The vial will be warm.

Note: *Proper mixing is important for complete recovery. Hold the vial vertical with the cap up. Invert the vial and wait for all of the solution to flow to the cap end. Pause. Return the vial to the upright position and wait for all of the solution to flow to the vial bottom. This is one inversion (10 inversions = 30 seconds).*



14. The display will show: **05:00 Timer 3**

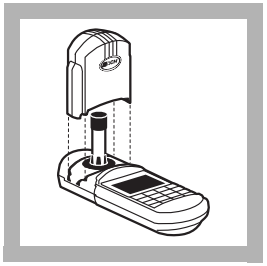
Press: **ENTER**

A five-minute reaction period will begin. Do not invert the vial again.

Note: *The yellow color will intensify.*

15. Insert the COD/ TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



16. When the timer beeps, wipe the outside of the Total Nitrogen Reagent C vial containing the reagent blank.

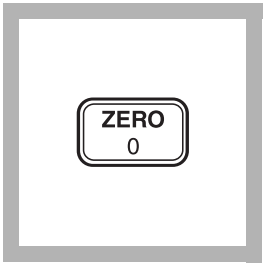
Place the vial into the adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

Tightly cover the vial with the instrument cap.

Note: *Do not move the vial from side to side during insertion, as this can cause errors.*

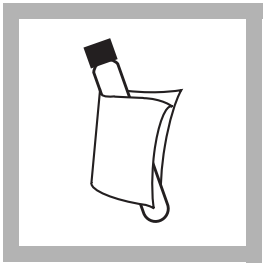
Note: *Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*



17. Press: **ZERO**

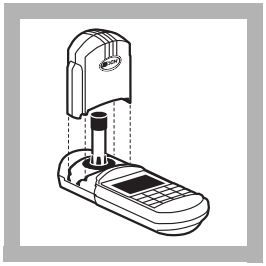
The cursor will move to the right, then the display will show:

0 mg/L N



18. Wipe the Total Nitrogen Reagent C vial containing the sample.

Note: *Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*



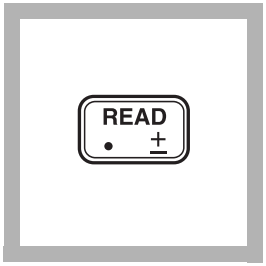
19. Place the vial into the adapter with the Hach logo facing the front of the instrument.

Push straight down on the top of the vial until it seats solidly into the adapter.

Tightly cover the vial with the instrument cap.

Note: *Do not move the vial from side to side during insertion, as this can cause errors.*

Note: *Multiple samples may be read after zeroing on one reagent blank.*



20. Press: **READ**

The cursor will move to the right, then the result in mg/L nitrogen (N) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see* Standard Adjust *in* Section1 *of the Procedures Manual).*

Note: *If the display flashes* **Limit***, dilute the sample and repeat the digestion and the colorimetric finish. The digestion must be repeated for accurate results; diluting and repeating the color finish does not yield complete results. Multiply the result by the dilution factor; see SECTION 1.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Best results are obtained with immediate analysis.

Preserve the sample by reducing the pH to 2 or less with concentrated sulfuric acid (at least 2 mL/L). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Correcting for Volume Additions* in *Section 1*.

Accuracy Check

This method generally yields 95-100% recovery on organic nitrogen standards. For proof of accuracy Hach offers a set of three Primary Standards for Kjeldahl Nitrogen.

1. Prepare one or more of the following three solutions. Each preparation is for an equivalent 120 mg/L N standard. Use water that is free of all organic and nitrogen-containing species.

   a) Weigh 1.6208 g of Ammonium p-Toluenesulfonate (PTSA). Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   b) Weigh 2.1179 g of Glycine p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

   c) Weigh 2.5295 g of Nicotinic p-Toluenesulfonate. Dissolve in a 1000-mL volumetric flask with deionized water. Add deionized water to the 1000-mL mark.

2. Analyze each of these solutions using the test procedure above. Calculate the percent recovery for each using this formula:

$$\% \text{ recovery} = \frac{\text{measured concentration}}{120} \times 100$$

The percent recovery should be:

Compound	Lowest Expected % Recovery
Ammonia-PTSA	95%
Glycine-PTSA	95%
Nicotinic-PTSA	95%

Hach analysts have found Ammonia-PTSA to be the most difficult to digest. Other compounds may yield different percent recoveries.

Standard Solution Method

For proof of accuracy, substitute 0.5 mL of a 125 mg/L ammonia nitrogen standard solution for the sample in the procedure. To prepare a 125-mg/L standard, use a 25-mL Class A pipet to transfer 25.00 mL of a 1000-mg/L Ammonia Nitrogen Standard

(see *OPTIONAL REAGENTS* on page *400*) to a 200-mL Class A volumetric flask. Dilute to the line with organic-free water.

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off an Ammonia Nitrogen Voluette™ Ampule Standard Solution, 1000 mg/L as NH_3-N.

c) Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to the three mixing cylinders.

d) Stopper each cylinder and mix thoroughly.

e) Add 0.5 mL of each prepared solution, respectively, to three HR Total Nitrogen Hydroxide Digestion Vials.

f) Analyze each standard addition sample as described in the procedure. The nitrogen concentration should increase 4 mg/L N for each 0.1 mL of standard added.

g) If these increases do not occur, see *Standard Additions* in *Section 1* for troubleshooting information.

Blanks for Colorimetric Measurement

The reagent blank may be used repeatedly for measurements using the same lots of reagents. Store the reagent blank in the dark at room temperature (18–25 °C) for a maximum of seven days. If a small amount of white floc appears prior to the end of one week, discard the reagent blank and prepare a new one.

Method Performance

Precision

In a single laboratory, using a standard solution of 125 mg/L N and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than 3 mg/L N. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 69 is 7 mg/L N. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering substances that resulted in a concentration change of ±10%:

Substance	Level and Effect
Bromide	> 240 ppm; positive interference
Chloride	≥3000 ppm; positive interference

The substances in the following table have been tested and found **not** to interfere up to the indicated levels:

Substance	Maximum Level Tested (mg/L)
Barium	10.4
Calcium	1200
Chromium (3+)	2
Iron	8
Lead	26.4 ppb
Magnesium	2000
Organic Carbon	600
pH	13 pH units
Phosphorus	400
Silica	600
Silver	3.6
Tin	6.0

The large amounts of nitrogen-free organic compounds in some samples may decrease digestion efficiency by consuming some of the persulfate reagent. Samples known to contain high levels of organics should be diluted and re-run to verify digestion efficiency.

Summary of Method

An alkaline persulfate digestion converts all forms of nitrogen to nitrate. Sodium metabisulfite is added after the digestion to eliminate halogen oxide interferences. Nitrate then reacts with chromotropic acid under strongly acidic conditions to form a yellow complex with an absorbance maximum near 420 nm.

REQUIRED REAGENTS

Test 'N Tube HR Total Nitrogen Reagent Set (50 vials) 27141-00
Includes: (1) 26718-46, (1) 26719-46, (1) 26720-46, *(50) Hydroxide Digestion Vials, *(50) Acid Solution Vials

Description	Quantity Required Per Test	Unit	Cat. No.
HR Total Nitrogen Hydroxide Digestion Vials	1 vial	50/pkg	*
Total Nitrogen Persulfate Reagent Powder Pillows	1 pillow	50/pkg	26718-46
Total Nitrogen Reagent A, Bisulfite Powder Pillows	1 pillow	50/pkg	26719-46
Total Nitrogen Reagent B, Indicator Powder Pillows	1 pillow	50/pkg	26720-46
Total Nitrogen Reagent C Vials, Acid Solution	1 vial	50/pkg	*

REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
COD/TNT Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips for 19700-01	2	50/pkg	21856-96
Test Tube Rack, for cooling vials	1-3	each	18641-00

OPTIONAL REAGENTS

Nitrogen, Ammonia, 1000 mg/L NH_3-N	1 L	23541-53
Nitrogen, Ammonia, Voluette Ampule, 1000 mg/L NH_3-N, 10 mL	16/pkg	23541-10
Sulfuric Acid, ACS	500 mL	979-49
Primary Standards for Kjeldahl Nitrogen	set of 3	22778-00
Ammonium p-Toluenesulfonate	25 g	22779-24
Glycine p-Toluenesulfonate	25 g	22780-24
Nicotinic Acid p-Toluenesulfonate	25 g	22781-24
Sodium Hydroxide Standard Solution, 5.0 N	50 mL MDB	2450-26
Wastewater Influent Standard, Inorganics (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49
Water, organic-free	500 mL	26415-49

* These items are not sold separately. Please order the complete set (Cat. No. 27141-00) as a replacement.

OPTIONAL APPARATUS

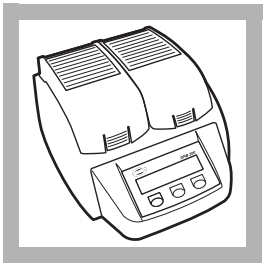
Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Balance, analytical, 115 Vac	each	28014-01
Balance, analytical, 230 Vac	each	28014-02
Cots, finger	2/pkg	14647-02
Cylinder, graduated, mixing, 25 mL	3 each	26363-40
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm		LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm		LTV082.52.30001
Flask, volumetric, Class A, 1000 mL	3 each	14574-53
Flask, volumetric, Class A, 200 mL	each	14574-45
Pipet, volumetric, Class A, 25 mL	2 each	14515-40
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224. Out side the U.S.A— Contact the Hach office or distributor serving you.
Outside the U.S.A.—Contact the Hach office or distributor serving you.

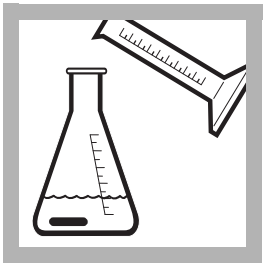
ORGANIC CARBON, TOTAL, Low Range (0.0–20.0 mg/L C)

Direct Method* **For water, drinking water, and wastewater**

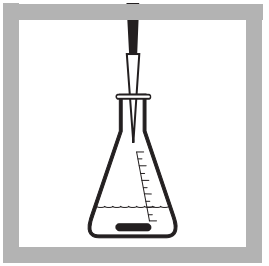


1. Turn on the DRB 200 reactor. Heat to 103-105 °C.

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*

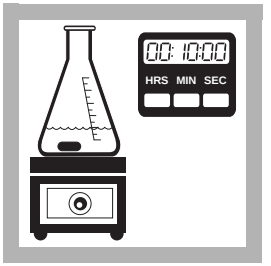


2. Use a graduated cylinder to add 10 mL of sample to a 50-mL erlenmeyer flask containing a stir bar.

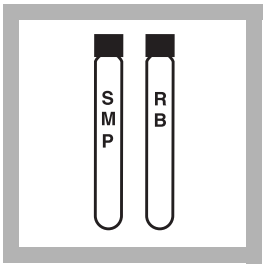


3. Add 0.4 mL of Buffer Solution, pH 2.0.

Note: *Use pH paper to make sure the sample pH is 2.*

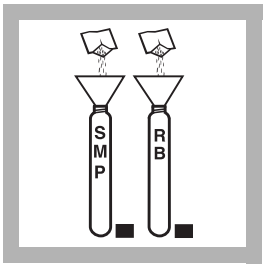


4. Place the flask on a stir plate and stir at a moderate speed for 10 minutes.

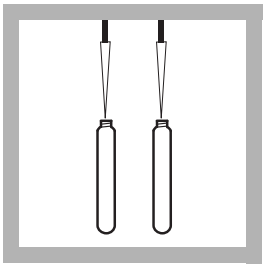


5. Label two Low Range Acid Digestion vials: **sample** and **reagent blank**.

Note: *A reagent blank is required for each series of samples.*



6. Using a funnel, add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).

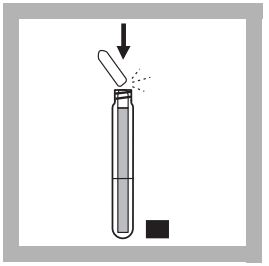


7. Use a TenSette® Pipet to add 3.0 mL of **organic-free water** to the **reagent blank** vial and 3.0 mL of **prepared sample** to the **sample** vial. Swirl to mix.

8. Rinse two blue Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

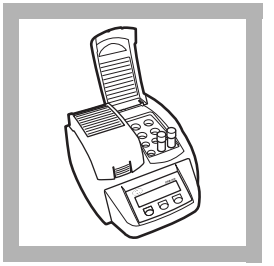
Note: Do not touch the ampules on the sides after wiping. Pick them up by the top.

* Patent pending

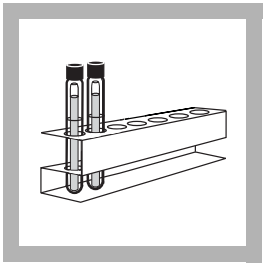


9. Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

Note: Do not invert or tilt the vial after inserting the ampule to prevent the Indicator Reagent from mixing with the contents of the acid digestion vial.

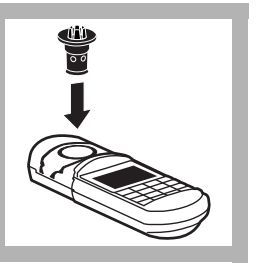


10. Cap the vial assemblies tightly and place them in the reactor for 2 hours at 103-105 °C.



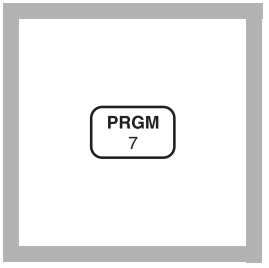
11. Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for **one hour** for accurate results.



12. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

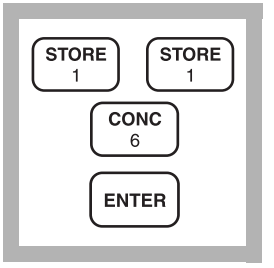
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



13. Enter the stored program number for Low Range TOC.

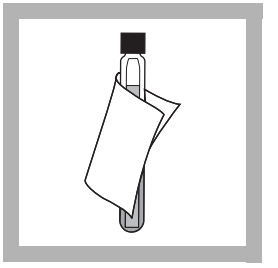
Press: **PRGM**

The display will show:
PRGM?



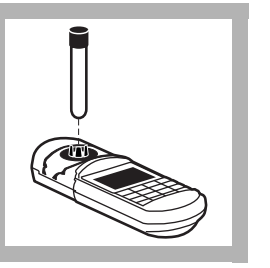
14. Press: **116 ENTER**

The display will show **mg/L** and the **ZERO** icon.



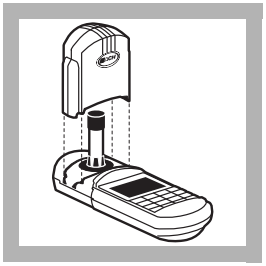
15. Wipe the reagent blank vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

Note: *The liquid in the reagent blank vial should be dark blue.*

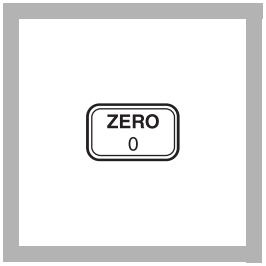


16. Place the **reagent blank** vial assembly in the adapter.

Push straight down on the top of the vial until it seats solidly in the adapter.



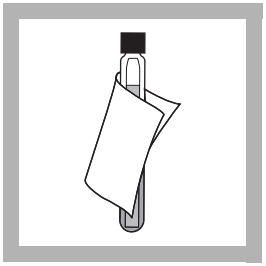
17. Tightly cover the vial assembly with the instrument cap.



18. Press: **ZERO**

The cursor will move to the right, then the display will show:

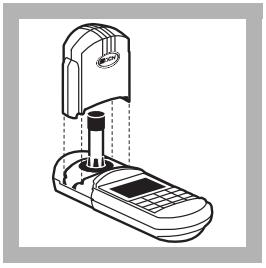
0.0 mg/L C



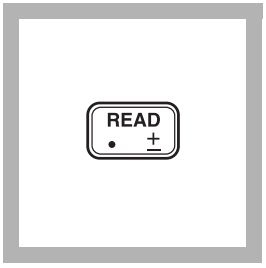
19. Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

20. Place the **sample** vial assembly in the adapter.

Push straight down on the top of the vial assembly until it seats solidly in the adapter.



21. Tightly cover the vial assembly with the instrument cap.



22. Press: **READ**

The cursor will move to the right, then the result in mg/L C will be displayed.

Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

Accuracy Check

Standard Solutions Method

a. Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.

Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).

b. Prepare a 10.0 mg/L C standard by transferring 1.00 mL of the stock standard to a 100-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh daily.

Standard Additions Method

a. Prepare a 150 mg/L C standard by transferring 15.00 mL of 1000 mg/L C stock solution to a 100-mL Class A volumetric flask. Dilute to volume with organic-free water. Mix.

b. Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 150 mg/L C standard to each of three Acid Digestion vials.

c. Add the contents of one TOC Persulfate powder pillow to each vial.

d. Add 3.0 mL of sample to each vial. Swirl to mix.

e. Proceed with the procedure starting at *step 8*.

f. The mg/L C concentration should increase by 5.0 mg/L for each 0.1 mL increment.

Method Performance

Precision

In a single laboratory, using a standard solution of 9.0 mg/L C and one lot of reagents, a single operator obtained a standard deviation of ±0.5 mg/L C.

Estimated Detection Limit

The estimated detection limit for Method 10129 is 0.3 mg/L C.

Sensitivity

At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 0.2 mg/L C.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br^-
Bromine	25 mg/L Br_2
Calcium	2000 mg/L as $CaCO_3$
Chloride	500 mg/L
Chlorine	10 mg/L Cl_2
Chlorine Dioxide	6 mg/L ClO_2
Copper	10 mg/L
Cyanide	10 mg/L CN^-
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as $CaCO_3$
Manganese (VII)	1 mg/L
Monochloramine	14 mg/L NH_2Cl as Cl_2
Nitrite	500 mg/L NO_2^-
Ozone	2 mg/L O_3
Phosphate	3390 mg/L PO_4^{2-}

Table 1 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Silica	100 mg/L SiO_2
Sulfate	5000 mg/L SO_4^{2-}
Sulfide	20 mg/L S^{2-}
Sulfite	50 mg/L SO_3^{2-}
Zinc	5 mg/L

If the sample contains greater than 600 mg/L $CaCO_3$ alkalinity, lower the sample pH to less than 7 before testing by adding sulfuric acid solution.

Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 50 NTU have been tested without interference.

Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

REQUIRED REAGENTS

Description	Qty/Test	Unit	Cat. No.
Total Organic Carbon Direct Method Low Range Test 'N Tube Reagent Set	50	vials	27603-45
Includes:			
Acid Digestion Solution Vials, Low Range TOC	1	50/pkg	*
Buffer Solution, Sulfate	0.4 mL	25 mL	452-33
Funnel, micro	1	each	25843-35
Indicator Ampules, Low Range TOC	1	10/pkg	*
TOC Persulfate Powder Pillows	1	50/pkg	*
Water, organic-free**	3.0 mL	500 mL	26415-49

* These items are not sold separately.

** This item must be purchased separately.

REQUIRED APPARATUS

Description	Qty/Test	Unit	Cat. No.
Cylinder, graduated, 10-mL	1	each	508-38
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Flask, Erlenmeyer, 50-mL	1	each	505-41
Magnetic Stirrer, 115 V, 4" x 4"	1	each	28812-00
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for 19700-01 TenSette® Pipet	2	50/pkg	21856-96
Pipet Tips, for 19700-10 TenSette® Pipet	2	50/pkg	21997-96
Stir Bar, Magnetic	1	each	45315-00
Wipes, Disposable, Kimwipes	1	280/pkg	20970-00

OPTIONAL REAGENTS

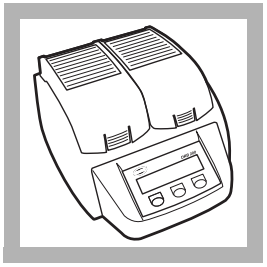
Potassium Acid Phthalate		500 g	315-34
Sulfuric Acid Reagent Solution, 5.25 N		100 mL MDB	2449-32
TOC Standard Solution Ampules (KHP Standard, 1000 mg/L C)		5/pkg	27915-05
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28332-49

OPTIONAL APPARATUS

Analytical Balance		each	28014-01
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm			LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm			LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm			LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm			LTV082.52.30001
Flask, volumetric, 100-mL		each	14574-42
Pipet, Class A, 200-mL		each	14515-35
Pipet, Class A, 15.00-mL		each	14515-39

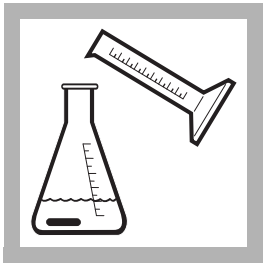
ORGANIC CARBON, TOTAL, Mid Range

Direct Method* **For wastewater and industrial waters**

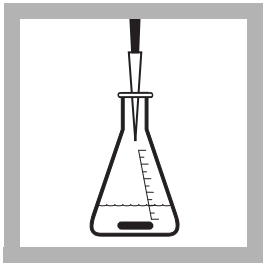


1. Turn on the DRB 200 reactor. Heat to 103–105 °C.

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*

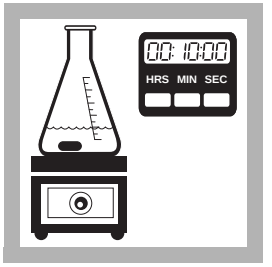


2. Use a graduated cylinder to add 10 mL of sample to a 50-mL erlenmeyer flask containing a stir bar.

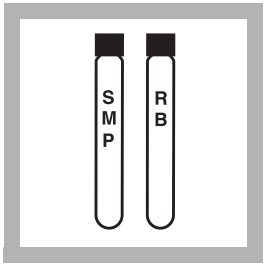


3. Add 0.4 mL of Buffer Solution, pH 2.0.

Note: *Use pH paper to make sure the sample pH is 2.*

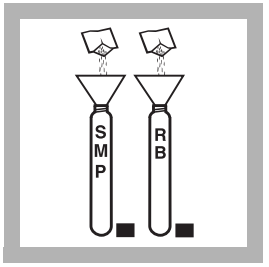


4. Place the flask on a stir plate and stir at a moderate speed for 10 minutes.

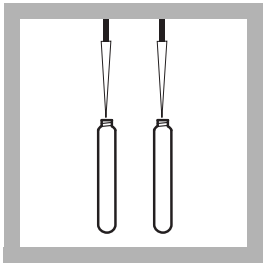


5. Label two Mid Range Acid Digestion vials: **sample** and **reagent blank**.

Note: *A reagent blank is required for each series of samples.*



6. Using a funnel, add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).

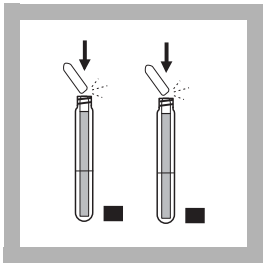


7. Use a TenSette® Pipet to add 1.0 mL of **organic-free water** to the **reagent blank** vial and1.0 mL of **prepared sample** to the **sample** vial. Do not cap the vial; swirl gently to mix.

8. Rinse two blue Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

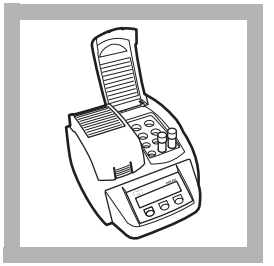
Note: Do not touch the ampules on the sides after wiping. Pick them up by the top.

* Patent pending

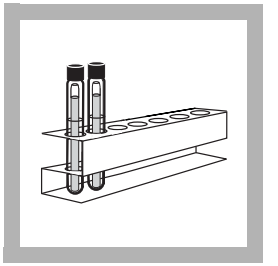


9. Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

Note: Do not invert or tilt the vial after inserting the ampule to prevent the Indicator Reagent from mixing with the contents of the acid digestion vial.

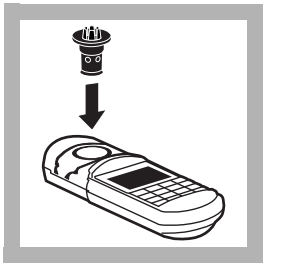


10. Cap the vial assemblies tightly and place them in the reactor for 2 hours at 103–105 °C.



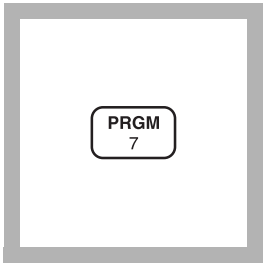
11. Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for **one hour** for accurate results.



12. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

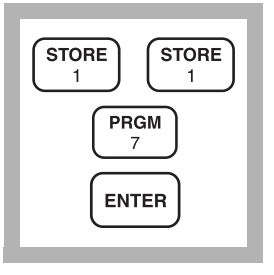
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



13. Enter the stored program number for Mid Range TOC.

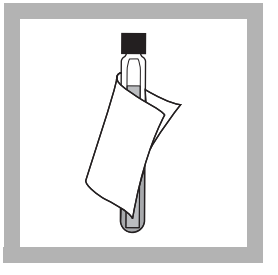
Press: **PRGM**

The display will show:
PRGM?



14. Press: **117 ENTER**

The display will show **mg/L** and the **ZERO** icon.

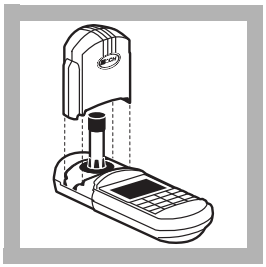


15. Wipe the reagent blank vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

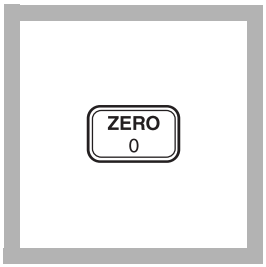
Note: *The liquid in the reagent blank vial should be dark blue.*

16. Place the **reagent blank** vial assembly in the adapter.

Push straight down on the top of the vial until it seats solidly in the adapter.



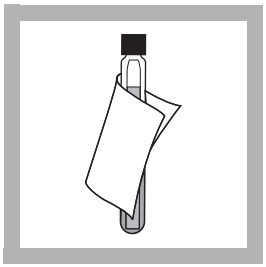
17. Tightly cover the vial assembly with the instrument cap.



18. Press: **ZERO**

The cursor will move to the right, then the display will show:

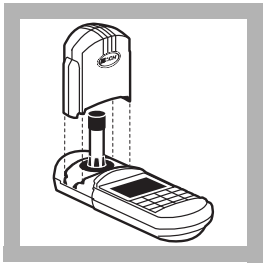
0 mg/L C



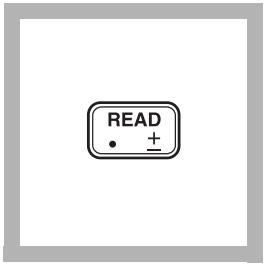
19. Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

20. Place the **sample** vial assembly in the adapter.

Push straight down on the top of the vial assembly until it seats solidly in the adapter.



21. Tightly cover the vial assembly with the instrument cap.



22. Press: **READ**

The cursor will move to the right, then the result in mg/L C will be displayed.

Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

Accuracy Check

Standard Solutions Method

a. Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature. Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).

b. Prepare a 100 mg/L C standard by transferring 5.00 mL of the stock standard to a 50-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh weekly.

Standard Additions Method

a. Prepare a 300 mg/L C standard by transferring 15.00 mL of 1000 mg/L C stock solution to a 50-mL Class A volumetric flask. Dilute to volume with Organic-Free Water. Mix.

b. Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 300 mg/L C standard to each of three Acid Digestion vials.

c. Add the contents of one TOC Persulfate powder pillow to each vial.

d. Add 1.0 mL of sample to each vial. Swirl to mix.

e. Proceed with the procedure starting at *step 8*.

f. The mg/L C concentration should increase by 30 mg/L for each 0.1 mL increment.

Method Performance

Precision

mg/L C	95% Confidence Limits
15	± 5 mg/L C
50	± 6 mg/L
75	± 7 mg/L
115	± 4 mg/L
150	± 6 mg/L

Estimated Detection Limit

Use Method Number 10173 to test TOC levels below 15 mg/L C.

Sensitivity

At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 1.9 mg/L C.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br
Bromine	25 mg/L Br_2
Calcium	2000 mg/L as $CaCO_3$
Chloride	1500 mg/L
Chlorine	10 mg/L Cl_2
Chlorine Dioxide	6 mg/L ClO_2
Copper	10 mg/L
Cyanide	10 mg/L CN
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as $CaCO_3$
Manganese (VII)	1 mg/L

Table 1 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Monochloramine	14 mg/L NH_2Cl as Cl_2
Nitrite	500 mg/L NO_2^-
Ozone	2 mg/L O_3
Phosphate	3390 mg/L PO_4^{3-}
Silica	100 mg/L SiO_2
Sulfate	5000 mg/L SO_4^{2-}
Sulfide	20 mg/L S^{2-}
Sulfite	50 mg/L SO_3^{2-}
Zinc	5 mg/L

Note: *If the sample contains greater than 1000 mg/L $CaCO_3$ alkalinity, lower the sample pH to less than 7 before testing by adding sulfuric acid solution.*

Note: *Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 50 NTU have been tested without interference.*

Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

Instrument Setup

This procedure will add the current method as a new Hach program to your DR/850 or DR/890.

1. Turn the instrument on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key until the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change a number already entered.

Line Number	Entry	Line Number	Entry
1	117	29	0
2	42	30	0
3	72	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	66	36	0
9	36	37	0
10	92	38	0
11	40	39	0
12	195	40	0
13	89	41	0
14	74	42	0
15	61	43	0
16	0	44	165
17	0	45	128
18	0	46	0
19	0	47	10
20	67	48	0
21	0	49	0
22	0	50	0
23	0	51	0
24	0	52	0
25	0	53	0
26	0	54	25
27	0	55	0
28	0	56	255

REQUIRED REAGENTS

Total Organic Carbon Direct Method Mid Range
Test 'N Tube Reagent Set ..... 50 vials 28159-45

Includes:

Description	Quantity Required Per Test	Unit	Cat. No.
Acid Digestion Solution Vials, Mid Range TOC	1	50/pkg	*
Buffer Solution, Sulfate	0.4 mL	25 mL	452-33
Funnel, micro	1	each	25843-35
Indicator Ampules, Mid/High Range TOC	1	50/pkg	*
TOC Persulfate Powder Pillows	1	50/pkg	*
Water, organic-free**	1.0 mL	500 mL	26415-49

REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Cylinder, graduated, 10-mL	1	each	508-38
Flask, Erlenmeyer, 50-mL	1	each	505-41
Magnetic Stirrer, 115 V, 4" x 4"	1	each	28812-00
Test Tube Rack	1–3	each	18641-00
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette® Pipet	2	50/pkg	21856-96
Stir Bar, Magnetic	1	each	45315-00
Wipes, Disposable, Kimwipes	1	280/pkg	20970-00

OPTIONAL REAGENTS

Description	Per Test	Unit	Cat. No.
TOC Standard Solution (KHP Standard, 1000 mg/L C)		5/pkg	27915-05
Potassium Acid Phthalate		500 g	315-34
Sulfuric Acid Reagent Solution, 5.25 N		100 mL MDB	2449-32

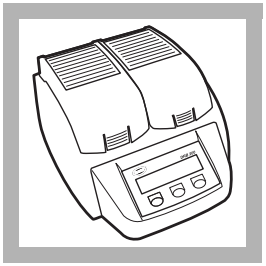
OPTIONAL APPARATUS

Analytical Balance		each	28014-01
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm			LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm			LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm			LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm			LTV082.52.30001
Flask, volumetric, 100-mL		each	14574-42
Pipet, Class A, 10.00-mL		each	14515-38
Pipet, Class A, 15.00-mL		each	14515-39
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28

* These items are not sold separately.
** This item must be purchased separately.

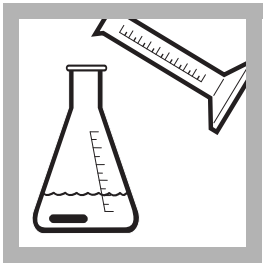
ORGANIC CARBON, TOTAL, High Range (20–700 mg/L C)

Direct Method* **For wastewater and industrial waters**

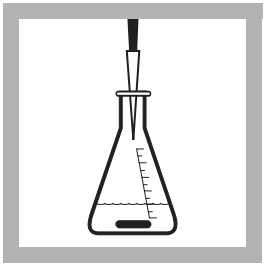


1. Turn on the DRB 200 reactor. Heat to 103-105 °C.

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*

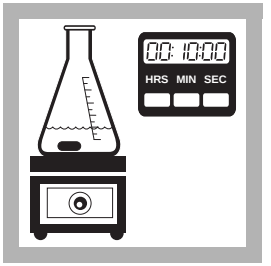


2. Use a graduated cylinder to add 10 mL of sample to a 50-mL erlenmeyer flask containing a stir bar.

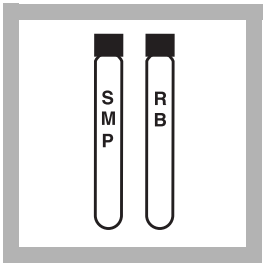


3. Add 0.4 mL of Buffer Solution, pH 2.0.

Note: *Use pH paper to make sure the sample pH is 2.*

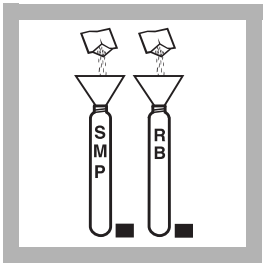


4. Place the flask on a stir plate and stir at a moderate speed for 10 minutes.

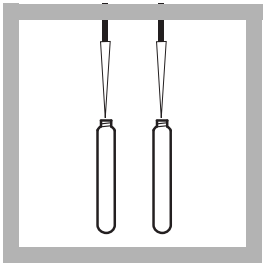


5. Label two High Range Acid Digestion vials: **sample** and **reagent blank**.

Note: *A reagent blank is required for each series of samples.*



6. Using a funnel, add the contents of one TOC Persulfate Powder Pillow to each Acid Digestion vial (colorless liquid).

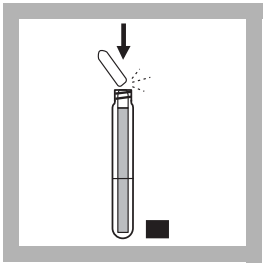


7. Use a TenSette® Pipet to add 0.3 mL of **organic-free water** to the **reagent blank** vial and 0.3 mL of **prepared sample** to the **sample** vial. Swirl to mix.

8. Rinse two blue Indicator Ampules with deionized water and wipe them with a soft, lint-free wipe.

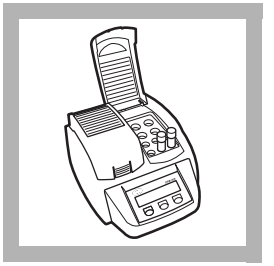
Note: Do not touch the ampules on the sides after wiping. Pick them up by the top.

* Patent pending

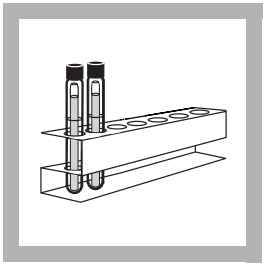


9. Lower one unopened ampule into each Acid Digestion vial. When the score mark on the ampule is level with the top of the Acid Digestion vial, snap the top off the ampule and allow it to drop into the Acid Digestion vial.

Note: Do not invert or tilt the vial after inserting the ampule to prevent the Indicator Reagent from mixing with the contents of the acid digestion vial.

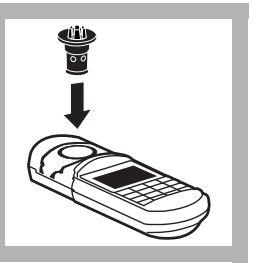


10. Cap the vial assemblies tightly and place them in the reactor for 2 hours at 103–105 °C.



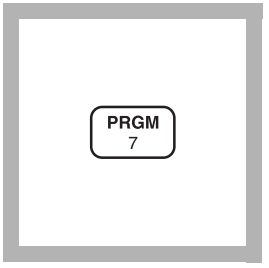
11. Carefully remove the vial assemblies from the reactor. Place them in a test tube rack.

Allow the vials to cool for **one hour** for accurate results.



12. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

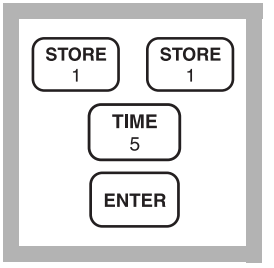
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



13. Enter the stored program number for High Range TOC.

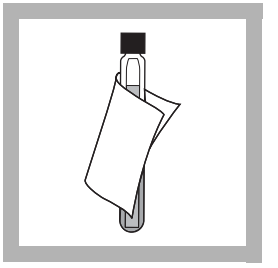
Press: **PRGM**

The display will show:
PRGM?



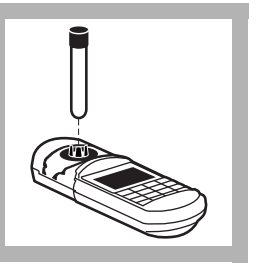
14. Press: **115 ENTER**

The display will show **mg/L** and the **ZERO** icon.



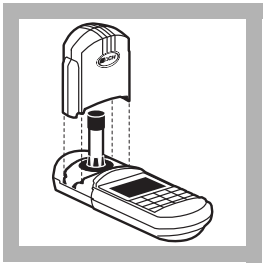
15. Wipe the reagent blank vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

Note: *The liquid in the reagent blank vial should be dark blue.*

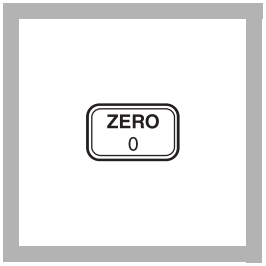


16. Place the **reagent blank** vial assembly in the adapter.

Push straight down on the top of the vial until it seats solidly in the adapter.



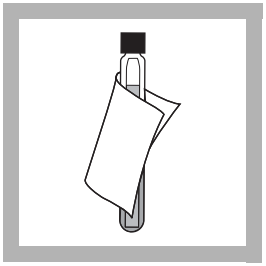
17. Tightly cover the vial assembly with the instrument cap.



18. Press: **ZERO**

The cursor will move to the right, then the display will show:

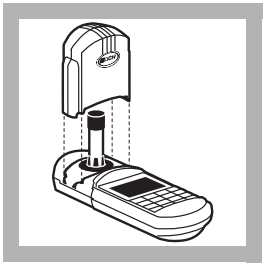
0 mg/L C



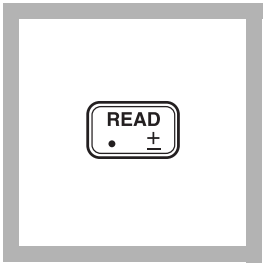
19. Wipe the sample vial assembly with a damp towel, followed by a dry one, to remove fingerprints or other marks.

20. Place the **sample** vial assembly in the adapter.

Push straight down on the top of the vial assembly until it seats solidly in the adapter.



21. Tightly cover the vial assembly with the instrument cap.



22. Press: **READ**

The cursor will move to the right, then the result in mg/L C will be displayed.

Sampling and Storage

Collect samples in clean glass bottles. Rinse the sample bottle several times with the sample to be collected. Fill the bottle with minimum headspace before capping. Test samples as soon as possible. Acid preservation is not recommended. Homogenize samples containing solids to assure representative samples.

Accuracy Check

Standard Solutions Method

a. Prepare a 1000 mg/L organic carbon stock standard by dissolving 2.1254 g dry primary standard Potassium Acid Phthalate in Organic-Free Reagent Water and dilute to 1000 mL. This stock standard is stable for about 1 month at room temperature.
Alternatively, open one ampule of TOC Standard Solution (Cat. No. 27915-05).

b. Prepare a 300 mg/L C standard by transferring 15.00 mL of the stock standard to a 50-mL Class A volumetric flask. Dilute to volume using Organic-Free Reagent Water. Stopper and mix thoroughly. Prepare this standard fresh weekly.

Standard Additions Method

a. Prepare a 300 mg/L C standard by transferring 18.00 mL of 1000 mg/L C stock solution to a 50-mL Class A volumetric flask. Dilute to volume with Organic-Free Water. Mix.

b. Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 300 mg/L C standard to each of three Acid Digestion vials.

c. Add the contents of one TOC Persulfate powder pillow to each vial.

d. Add 0.3 mL of sample to each vial. Swirl to mix.

e. Proceed with the procedure starting at *step 8*.

f. The mg/L C concentration should increase by 100 mg/L for each 0.1 mL increment.

Method Performance

Precision
In a single laboratory, using a standard solution of 360 mg/L C and one lot of reagents, a single operator obtained a standard deviation of ±8 mg/L C.

Estimated Detection Limit
Use Method Number 10129 to test TOC levels below 20 mg/L C.

Sensitivity
At mid-range, the sensitivity, expressed as the concentration change per 0.010 absorbance change, is 6 mg/L C.

Interferences

The following have been tested for interference and found not to interfere up to the indicated levels:

Table 1 Non-interfering Substances

Substance	Maximum Level Tested
Aluminum	10 mg/L
Ammonia Nitrogen	1000 mg/L as N
ASTM Wastewater	No effect
Bromide	500 mg/L Br
Bromine	25 mg/L Br_2
Calcium	2000 mg/L as $CaCO_3$
Chloride	5000 mg/L
Chlorine	10 mg/L Cl_2
Chlorine Dioxide	6 mg/L ClO_2
Copper	10 mg/L
Cyanide	10 mg/L CN
Iodide	50 mg/L
Iron (II)	10 mg/L
Iron (III)	10 mg/L
Magnesium	2000 mg/L as $CaCO_3$
Manganese (VII)	1 mg/L
Monochloramine	14 mg/L NH_2Cl as Cl_2
Nitrite	500 mg/L NO_2^-
Ozone	2 mg/L O_3
Phosphate	3390 mg/L PO_4^{3-}

Table 1 Non-interfering Substances (Continued)

Substance	Maximum Level Tested
Silica	100 mg/L SiO_2
Sulfate	5000 mg/L SO_4^{2-}
Sulfide	20 mg/L S^{2-}
Sulfite	50 mg/L SO_3^{2-}
Zinc	5 mg/L

If the sample contains greater than 600 mg/L $CaCO_3$ alkalinity, lower the sample pH to less than 7 before testing by adding sulfuric acid solution.

Most sample turbidity is either dissolved during the digestion stage or settled during the cooling period. Sample turbidities up to 900 NTU have been tested without interference.

Summary of Method

The total organic carbon (TOC) is determined by first sparging the sample under slightly acidic conditions to remove the inorganic carbon. In the outside vial, organic carbon in the sample is digested by persulfate and acid to form carbon dioxide. During digestion, the carbon dioxide diffuses into a pH indicator reagent in the inner ampule. The adsorption of carbon dioxide into the indicator forms carbonic acid. Carbonic acid changes the pH of the indicator solution which, in turn, changes the color. The amount of color change is related to the original amount of carbon present in the sample.

REQUIRED REAGENTS

Total Organic Carbon Direct Method High Range
Test 'N Tube Reagent Set ..... 50 vials 27604-45

Includes:

Description	Qty/Test	Unit	Cat. No.
Acid Digestion Solution Vials, High Range TOC	1	50/pkg	*
Buffer Solution, Sulfate	0.4 mL	25 mL	452-33
Funnel, micro	1	each	25843-35
Indicator Ampules, High Range TOC	1	10/pkg	*
TOC Persulfate Powder Pillows	1	50/pkg	*
Water, organic-free**	0.3 mL	500 mL	26415-49

* These items are not sold separately.

REQUIRED APPARATUS

Cylinder, graduated, 10-mL	1	each	508-38
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Flask, Erlenmeyer, 50-mL	1	each	505-41
Magnetic Stirrer, 115 V, 4" x 4"	1	each	28812-00
Safety Shield, laboratory bench	1	each	50030-00
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette®, 0.1 to 1.0 mL	1	each	19700-01
Pipet, TenSette®, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for 19700-01 TenSette® Pipet	2	50/pkg	21856-96
Pipet Tips, for 19700-10 TenSette® Pipet	2	50/pkg	21997-96
Stir Bar, Magnetic	1	each	45315-00
Wipes, Disposable, Kimwipes	1	280/pkg	20970-00

OPTIONAL REAGENTS

Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules		16/pkg	28335-10
Potassium Acid Phthalate		500 g	315-34
Sulfuric Acid Reagent Solution, 5.25 N		100 mL MDB	2449-32
TOC Standard Solution Ampules (KHP Standard, 1000 mg/L C)		5/pkg	27915-05
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28331-49

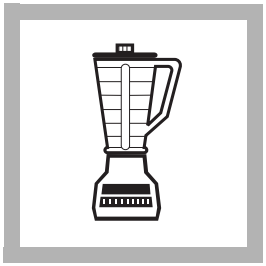
OPTIONAL APPARATUS

Analytical Balance		each	28014-01
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm			LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm			LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm			LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm			LTV082.52.30001
Flask, volumetric, 1000-mL		each	14574-53
Flask, volumetric, 100-mL		each	14574-42
Pipet, Class A, 10.00-mL		each	14515-38
Pipet, Class A, 15.00-mL		each	14515-39

** This item must be purchased separately.

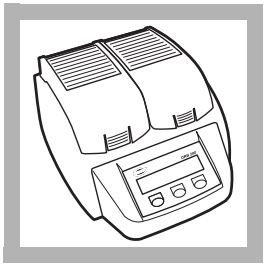
OXYGEN DEMAND, CHEMICAL

Reactor Digestion Method* USEPA approved for reporting wastewater analysis**
Digestion



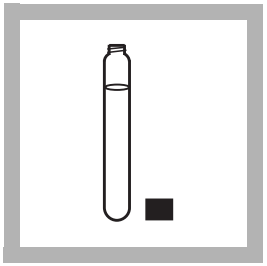
1. Homogenize 500 mL of sample for 2 minutes in a blender.

Note: For the 0-15,000 mg/L range, *homogenize 100 mL of sample. Pour the blended sample into a 250-mL beaker. Stir with a magnetic stirrer while withdrawing a sample aliquot. This improves accuracy and reproducibility.*



2. Turn on the DRB 200 Reactor. Preheat to 150 °C.

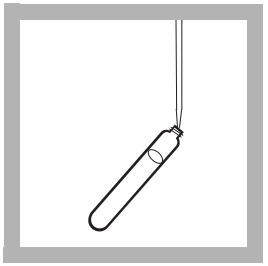
Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*



3. Remove the cap of a COD Digestion Reagent Vial for the appropriate range:

Sample Conc. Range (mg/L)	COD Digestion Reagent Vial Type
0 to 150	Low Range
0 to 1500	High Range
0 to 15,000	High Range Plus

Note: *The reagent mixture is light-sensitive. Keep unused vials in the opaque shipping container, in a refrigerator if possible. The light striking the vials during the test will not affect results.*



4. Hold the vial at a 45-degree angle. Pipet 2.00 mL (0.2 mL for the 0 to 15,000 mg/L range) of sample into the vial.

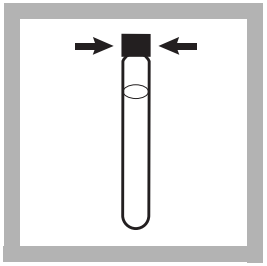
Note: For the 0-15,000 mg/L range, *pipet only 0.20 mL of sample, not 2.00 mL of sample, using a TenSette Pipet. For greater accuracy analyze a minimum of three replicates and average the results.*

Note: *Spilled reagent will affect test accuracy and is hazardous to skin and other materials. Do not run tests with vials which have been spilled. If spills occur, wash with running water.*

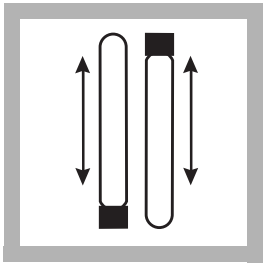
Caution: Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately or accidentally misused. Please read all warnings and the safety section of this manual. Wear appropriate eye protection and clothing. If contact occurs, flush the affected area with running water. Follow all instructions carefully.

* Jirka, A.M.; Carter, M.J. *Analytical Chemistry,* 1975, 47(8). 1397.

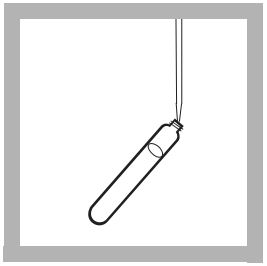
** *Federal Register,* April 21, 1980, 45(78), 26811-26812. The 0-15,000 mg/L range is **not** USEPA approved.



5. Replace the vial cap tightly. Rinse the outside of the COD vial with deionized water and wipe the vial clean with a paper towel.

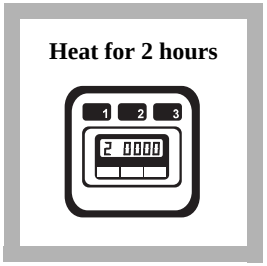


6. Hold the vial by the cap and over a sink. Invert gently several times to mix the contents. Place the vial in the preheated DRB 200 Reactor.
Note: *The vial will become very hot during mixing.*



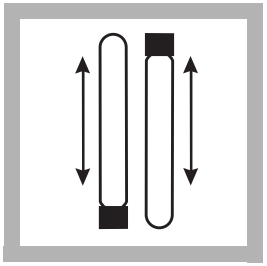
7. Prepare a blank by repeating Steps 3 to 6, substituting 2.00 mL (0.2 mL for the 0 to 15,000 mg/L range) deionized water for the sample.
Note: *Be sure the pipet is clean.*

Note: *One blank must be run with each set of samples. Run samples and blanks with vials from the same lot number (lot # is on the container label).*



8. Heat the vials for 2 hours.
Note: *Many samples are digested completely in less than two hours. If desired, measure the concentration (while still hot) at 15 minute intervals until the reading remains unchanged. Cool vials to room temperature for final measurement.*

9. Turn the reactor off. Wait about 20 minutes for the vials to cool to 120 °C or less.

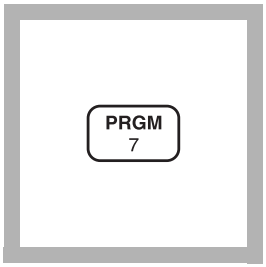


10. Invert each vial several times while still warm. Place the vials into a rack. Wait until the vials have cooled to room temperature.
Note: *If a pure green color appears in the reacted sample, measure the COD and, if necessary, repeat the test with a diluted sample.*

11. Use one of the following analytical techniques to measure the COD:

- Colorimetric method, 0-150 mg/L COD
- Colorimetric method, 0-1,500 mg/L COD
- Colorimetric method, 0-15,000 mg/L COD

Colorimetric Determination, 0 to 150 mg/L COD

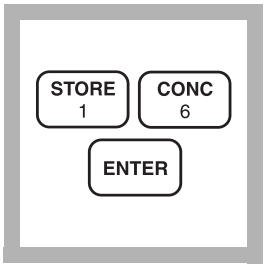


1. Enter the stored program number for chemical oxygen demand (COD), low range.

Press: **PRGM**

The display will show:

PRGM ?



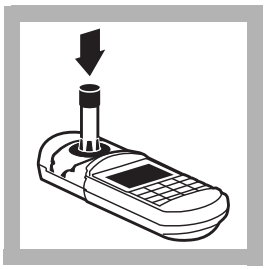
2. Press: **16 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon**.**

Note: *For alternate form (O_2), press the* **CONC** *key.*

3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*

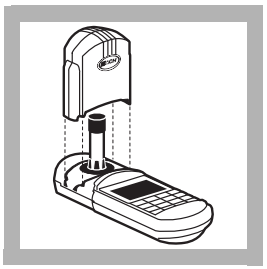
4. Clean the outside of the blank with a towel.
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



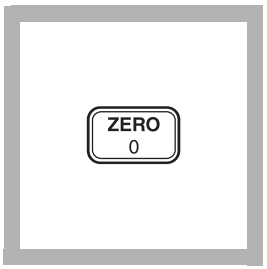
5. Place the blank in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



6. Tightly cover the vial with the instrument cap.
Note: *The blank is stable when stored in the dark. See Blanks for Colorimetric Determination following these procedures.*

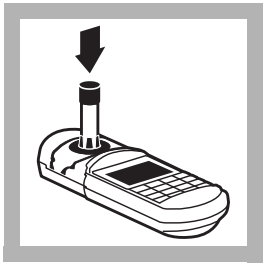


7. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L COD

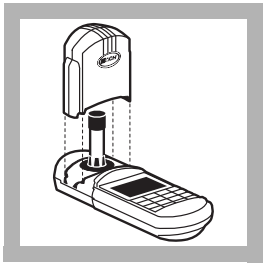
8. Clean the outside of the sample vial with a towel.



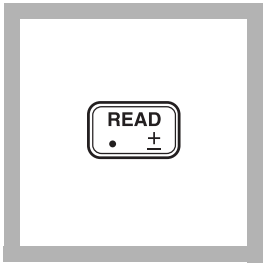
9. Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



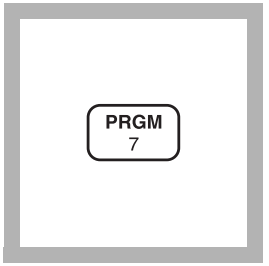
10. Tightly cover the vial with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

Colorimetric Determination, 0 to 1,500 and 0 to 15,000 mg/L COD

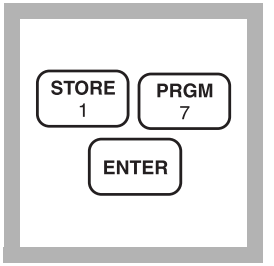


1. Enter the stored program number for chemical oxygen demand, high range.

Press: **PRGM**

The display will show:

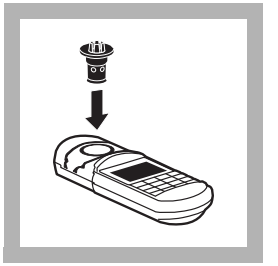
PRGM ?



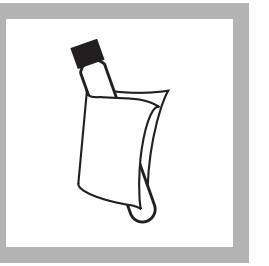
2. Press: **17 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon**.**

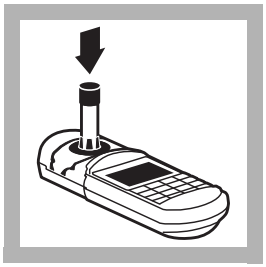
Note: *For alternate form (O_2), press the* **CONC** *key.*



3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.



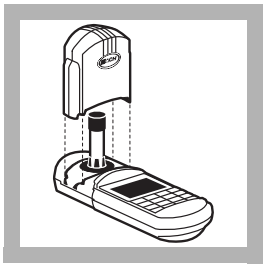
4. Clean the outside of the blank with a towel.
Note: *Wiping with a damp towel followed by a dry one will remove fingerprints or other marks.*



5. Place the blank in the adapter.

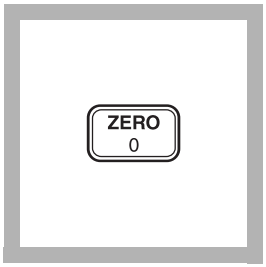
Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



6. Tightly cover the sample cell with the instrument cap.

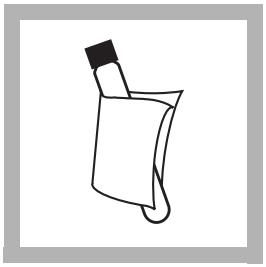
The blank is stable when stored in the dark. See Blanks for Colorimetric Determination following these procedures.



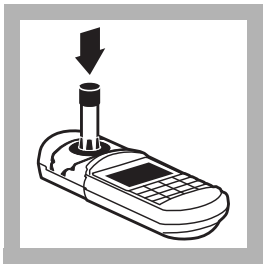
7. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L COD



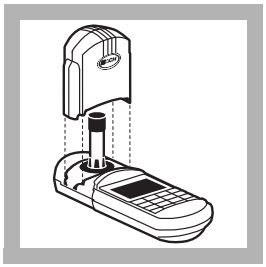
8. Clean the outside of the sample vial with a towel.



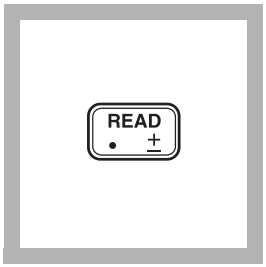
9. Place the sample in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



10. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

Note: *When using High Range Plus COD Digestion Reagent Vials, multiply the reading by 10.*

Note: *For most accurate results with samples near 1,500 or15,000 mg/L COD, repeat the analysis with a diluted sample.*

Sampling and Storage

Collect samples in glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C can be stored up to 28 days. Correct results for volume additions; see *Correction for Volume Additions* (Section 1) for more information.

Accuracy Check

Standard Solution Method

Check the accuracy of the 0 to 150 mg/L range with a 100 mg/L standard. Prepare by dissolving 85 mg of dried (120 °C, overnight) potassium acid phthalate (KHP) in 1 liter of deionized water. Use 2.0 mL as the sample volume. The expected result will be 100 mg/L COD. As an alternative, dilute 10 mL of 1000-mg/L COD Standard Solution to 100 mL to make a 100-mg/L standard.

Check the accuracy of the 0 to 1,500 mg/L range by using either a 300 mg/L or 1000 mg/L COD Standard Solution. Alternatively, prepare a 500 mg/L standard by dissolving 425 mg of dried (120 °C, overnight) KHP. Dilute to 1 liter with deionized water. Use 2.0 mL of one of these solutions as the sample volume.

Check the accuracy of the 0 to 15,000 mg/L range by using a 10,000 mg/L COD standard solution. Prepare the 10,000 mg/L solution by dissolving 8.500 g of dried (120 °C, overnight) KHP in 1 liter of deionized water. Use 0.2 mL of this solution as the sample volume; the expected result will be 10,000 mg/L COD.

Method Performance

Precision

Program #16: In a single laboratory, using a standard solution of 100 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±2 mg/L COD.

Program #17: In a single laboratory, using a standard solution of 1000 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ± 16 mg/L COD. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit (EDL)

The EDL for program 16 is 4 mg/L COD. The EDL for program 17 is 30 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Alternate reagents

Mercury-free COD2 Reagents can provide a mercury-free testing option for non-reporting purposes. For process control applications, COD2 reagents will eliminate mercury waste and save on disposal costs. These reagents are fully compatible with test procedures and calibration curves programmed into the DR/2400 spectrophotometer. Determine chloride and ammonia for accurate results.

Note: *Mercury-free COD2 reagents are not approved for USEPA reporting. Request a copy of the COD Reagent Vial Information Brochure, Lit. No. 1356, for more information about specific applications.*

Interferences

Chloride is the primary interference when determining COD concentration. Each COD vial contains mercuric sulfate that will eliminate chloride interference up to the level specified in column 1 in *Table 1*. Samples with higher chloride concentrations should be diluted. Dilute the sample enough to reduce the chloride concentration to the level given in column 2.

If sample dilution will cause the COD concentration to be too low for accurate determination, add 0.50 g of mercuric sulfate ($HgSO_4$) to each COD vial before the sample is added. The additional mercuric sulfate will raise the maximum chloride concentration allowable to the level given in column 3.

Table 1

	Column 1	**Column 2**	**Column 3**
Vial Type Used	Maximum Cl^- concentration in sample (mg/L)	Maximum Cl^- concentration of diluted samples (mg/L)	Maximum Cl^- concentration in sample when 0.50 $HgSO_4$ added (mg/L)
Low Range	2000	1000	8000
High Range	2000	1000	4000
High Range Plus	20,000	10,000	40,000

Blanks for Colorimetric Determination

The blank may be used repeatedly for measurements using the same lot of vials. Store it in the dark. Monitor decomposition by measuring the absorbance at the appropriate wavelength (420 or 610 nm). Zero the instrument in the absorbance mode, using a vial containing 5 mL of deionized water and measure the absorbance of the blank. Record the value. Prepare a new blank when the absorbance has changed by about 0.01 absorbance units.

Summary of Method

The mg/L COD results are defined as the mg of O_2 consumed per liter of sample under conditions of this procedure. In this procedure, the sample is heated for two hours with a strong oxidizing agent, potassium dichromate. Oxidizable organic compounds react, reducing the dichromate ion ($Cr_2O_7^{2-}$) to green chromic ion (Cr^{3+}). When the 0-150 mg/L colorimetric method is used, the amount of Cr^{6+} remaining is determined. When the 0-1,500 mg/L or 0-15,000 mg/L colorimetric method is used, the amount of Cr^{3+} produced is determined. The COD reagent also contains silver and mercury ions. Silver is a catalyst, and mercury is used to complex the chloride interference.

Pollution Prevention and Waste Management

Final samples will contain mercury (D009), silver (D011), and chromium (D007) at concentration levels regulated by the Federal RCRA. Please see *Section 3* for further information on proper disposal of these materials.

REQUIRED REAGENTS

Description	Qty/Test	Unit	Cat. No.
Select the appropriate COD Digestion Reagent Vial:			
Low Range, 0 to 150 mg/L COD	1 to 2 vials	25/pkg	21258-25
High Range, 0 to 1,500 mg/L COD	1 to 2 vials	25/pkg	21259-25
High Range Plus, 0 to 15,000 mg/L COD	1 to 2 vials	25/pkg	24159-25
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Blender, Osterizer, 120 V, 14 speed	1	each	26160-00
Blender, Osterizer, 240 V, 14 speed	1	each	26160-02
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001

REQUIRED APPARATUS (continued)

Description	Qty/Test	Unit	Cat. No.
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
COD/TNT Adapter	1	each	48464-00
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	1	50/pkg	21856-96
Pipet, volumetric, Class A, 2.00 mL	1	each	14515-36
Pipet Filler, safety bulb	1	each	14651-00
Test Tube Rack	1 to 2 racks	each	18641-00

ALTERNATE REAGENTS*

COD2, LR, 0 to 150 mg/L COD	1–2 vials	25/pkg	25650-25
COD2, HR, 0 to 1500 mg/L COD	1–2 vials	25/pkg	25651-25
COD2, HR, 0 to 1500 mg/L COD	1–2 vials	150/pkg	25651-15
COD2, HR, 0 to 15,000 mg/L COD	1–2 vials	25/pkg	28343-25

OPTIONAL REAGENTS

Description	Unit	Cat. No.
COD Digestion Reagent Vials, 0 to 150 mg/L COD	150/pkg	21258-15
COD Digestion Reagent Vials, 0 to 1,500 mg/L COD	150/pkg	21259-15
COD Standard Solution, 300 mg/L	200 mL	12186-29
COD Standard Solution, 1000 mg/L	200 mL	22539-29
Mercuric Sulfate	28.3 grams	1915-20
Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules	16/pkg	28335-10
Potassium Acid Phthalate, ACS	500 g	315-34
Potassium Dichromate Standard Solution, 0.25 N	1000 mL*	1809-53
Sulfuric Acid, ACS	500 mL**	979-49
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49

OPTIONAL APPARATUS

Balance, analytical, 115 V	each	28014-01
Balance, analytical, 230 V	each	28014-02
Beaker, 250 mL	each	500-46H
Cylinder, graduated, 5 mL	each	508-37
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm		LTV082.53.30001

* Mercury-free COD2 reagents are not approved for USEPA reporting. Request a copy of the COD Reagent Vial Information Brochure, Lit. No. 1356, for more information about specific applications.

** Contact Hach for larger sizes.

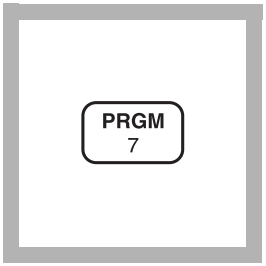
OPTIONAL APPARATUS (continued)

Description	Unit	Cat. No.
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm		LTV082.52.30001
Electromagnetic Stirrer, 120 V, with electrode stand	each	45300-01
Electromagnetic Stirrer, 230 V, with electrode stand	each	45300-02
Flask, volumetric, Class A, 1000 mL	each	14574-53
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
Pipet, serological, 5 mL	each	532-37
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 10 mL	each	14515-38
Spoon, measuring, 0.5 g	each	907-00
Stir Bar, 22.2 x 4.76 mm (7/8" x 3/16")	each	45315-00
Stir Bar Retriever	each	15232-00
Timer	each	26304-00

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

OXYGEN DEMAND, CHEMICAL (20 to 1,000 mg/L) For water and wastewater

Manganese III Digestion Method* (without chloride removal)



1. Enter the stored program number for Manganese III COD.

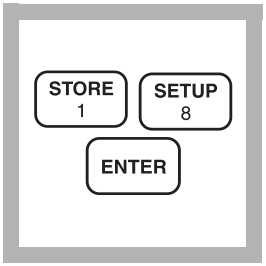
Press: **PRGM**

The display will show:

PRGM ?

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

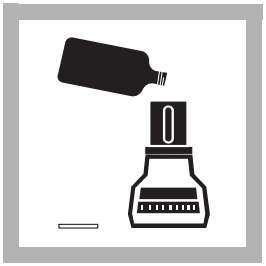
Note: *Preheat the COD Reactor to 150 °C for use later in the procedure.*



2. Press: **18 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon**.**

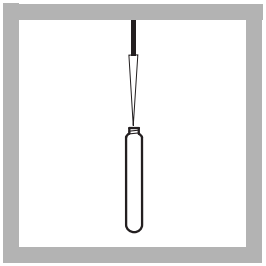
Note: *For alternate forms (O_2), press the* **CONC** *key.*



3. Homogenize 100 mL of sample for 30 seconds in a blender.

Note: *Blending promotes even distribution of solids and improves accuracy and reproducibility.*

Note: *Continue mixing the sample while pipetting if suspended solids are present.*



4. If chloride is not present in significant amounts†, pipet 0.50 mL of homogenized sample into a Mn III COD vial. Cap and invert several times to mix.

Note: *If the sample COD value is not between 20-1000 mg/L dilute the sample with deionized water to obtain a range of 20-1000 mg/L COD. Multiply the final result by the dilution factor.*

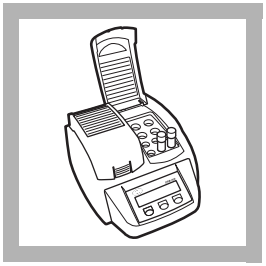
† To determine if chloride will interfere, run the sample with and without the chloride removal procedure and compare the results.

Caution: Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and the safety section of this manual. Wear appropriate eye protection and appropriate clothing. If contact occurs, flush the affected area with running water. Follow all instructions carefully.

* U.S. Patent 5,556,787

5. Prepare a blank (see note) by substituting 0.50 mL of deionized water for the sample. Continue with step 9 of this procedure.

Note: *The reagent blank is stable and can be reused. Verify reagent blank quality by measuring the absorbance of the blank vs. a clean COD vial filled with deionized water. The absorbance range should be about 1.36-1.43.*

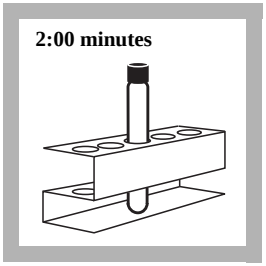


6. Place the vials in the DRB 200 Reactor that is preheated to 150 °C. Digest for 1 hour.

Note: *Boiling sample in the vials during digestion indicates the vial is not properly sealed; test results will be invalid.*

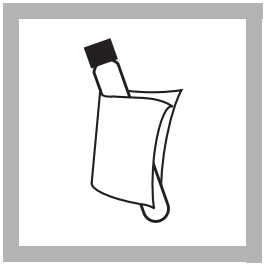
Note: *Samples can be digested up to 4 hours to oxidize more resistant organics. The prepared blank must be treated in the same manner.*

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*



7. Remove the vials and place them in a cooling rack for two minutes to air cool. Then cool the vials to room temperature in a cool water bath or running tap water. This usually takes about three minutes.

Note: *Occasionally a vial will develop a colorless upper layer and a purple lower layer. Invert the vial several times to mix and proceed. This will not affect test results.*

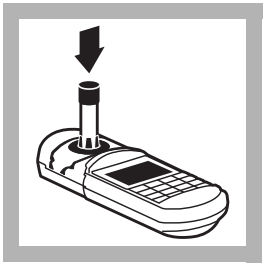


8. Remove the vials from the water and wipe with a clean, dry paper towel.

Invert the vials several times to mix.

9. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

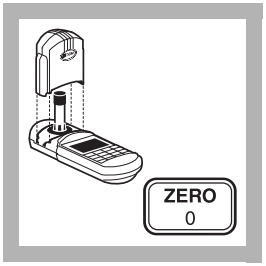
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



10. Place the blank in the sample cell adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



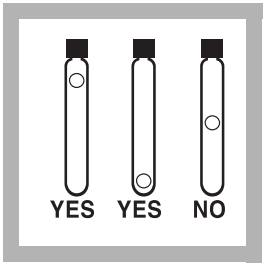
11. Tightly cover the sample cell with the instrument cap.

Note: *Clean the COD vial with a towel to remove fingerprints or other marks.*

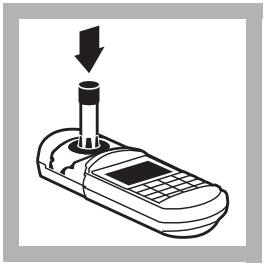
Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L COD



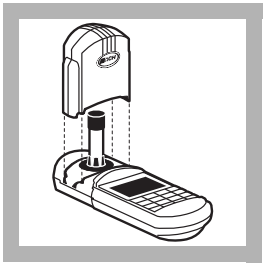
12. If the chloride removal was done, make sure the filter disc is not suspended in the middle of the vial; it can interfere with the instrument reading. Move it with gentle swirling or by lightly tapping the vial on the table top.



13. Place the sample in the adapter.

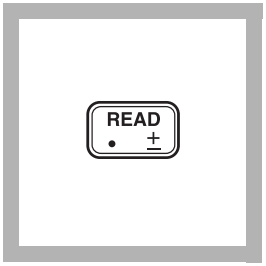
Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



14. Tightly cover the sample cell with the instrument cap.

Note: *Clean the COD vial with a towel to remove fingerprints or other marks.*



15. Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Note: *Adjust the result for any sample dilution in Steps 4 or 6.*

Sampling and Storage

Collect samples in clean glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with concentrated sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C may be stored up to 28 days. Correct results for volume additions; see *Correcting for Volume Additions* (*Section 1*) for more information.

Accuracy Check

Standard Solution Method

Prepare an 800 mg/L COD standard solution by adding 0.6808 g of
dried (103 °C, overnight) potassium acid phthalate (KHP) to 1 liter of deionized water. Use 0.50 mL of this solution (0.60 mL for the chloride removal procedure) as the sample volume. The result should be
800 ±26 mg/L COD.

An 800 mg/L COD solution can also be purchased directly from Hach (see *Optional Reagents*).

Method Performance (for Manganic III COD without the chloride removal procedure)

Precision

In a single laboratory, using a standard solution of 800 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±23 mg/L COD.

Estimated Detection Limit (EDL)

The EDL for program 18 is 14 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Inorganic materials may also be oxidized by trivalent manganese and constitute a positive interference when present in significant amounts. Chloride is the most common interference and is removed by sample pretreatment with the Chloride Removal Cartridge. If chloride is known to be absent or present in insignificant levels, the pretreatment can be omitted. A simple way to determine if chloride will affect test results is to run

routine samples with and without the chloride removal, then compare results. Other inorganic interferences (i.e., nitrite, ferrous iron, sulfide) are not usually present in significant amounts. If necessary, these interferences can be corrected for after determining their concentrations with separate methods and adjusting the final COD test
results accordingly.

Ammonia nitrogen is known to interfere in the presence of chloride; it does not interfere if chloride is absent.

Summary of Method

Chemical oxygen demand (COD) is defined as "... a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant" (APHA Standard Methods, 19th ed., 1995). Trivalent manganese is a strong, non-carcinogenic chemical oxidant that changes quantitatively from purple to colorless when it reacts with organic matter. It typically oxidizes about 80% of the organic compounds. Studies have shown that the reactions are highly reproducible and test results correlate closely to Biochemical Oxygen Demand (BOD) values and hexavalent chromium COD tests. None of the oxygen demand tests provide 100% oxidation of all
organic compounds.

A calibration is provided which is based on the oxidation of Potassium Acid Phthalate (KHP). A different response may be seen in analyzing various wastewaters. The KHP calibration is adequate for most applications. The highest degree of accuracy is obtained when test results are correlated to a standard reference method such as BOD or one of the chromium COD methods. Special waste streams or classes will require a separate calibration to obtain a direct mg/L COD reading or to generate a correction factor for the precalibrated KHP response. The sample digestion time can be extended up to 4 hours for samples which are difficult to oxidize.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Manganese III COD Reagent Vials, 20-1000 mg/L	1	25/pkg	26234-25
Sulfuric Acid, concentrated	1 mL	4 Kg	979-09
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Adapter, COD/TNT	1	each	48464-00
Blender, Osterizer, 120 Vac, 14-speed	1	each	26747-00
Blender Container, 118 mL	1	2/pkg	26748-00
Cap, with inert Teflon liner, for mixing bottle	varies	12/pkg	24018-12
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Forceps, extra fine point	1	each	26696-00
Mixing Bottle, glass, for sample + acid	1	each	24276-06
Pipet, TenSette, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette	2	250/pkg	21997-25
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette	2	1000/pkg	21856-28
Test Tube Rack, stainless steel	1	each	18641-00

OPTIONAL REAGENTS

COD Standard Solution, 800 mg/L COD		200 mL	26726-29
Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules		16/pkg	28335-10
Potassium Acid Phthalate		500 g	315-34
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28332-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28331-49

OPTIONAL APPARATUS

Dispenser for sulfuric acid		each	25631-37
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm			LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm			LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm			LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm			LTV082.52.30001

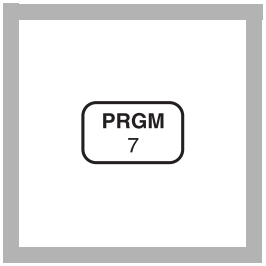
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

OXYGEN DEMAND, CHEMICAL (20 to 1,000 mg/L) For water and wastewater

Manganese III Digestion Method* (with chloride removal)



1. Enter the stored program number for Manganese III COD.

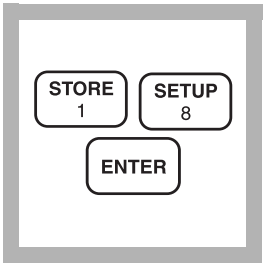
Press: **PRGM**

The display will show:

PRGM ?

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

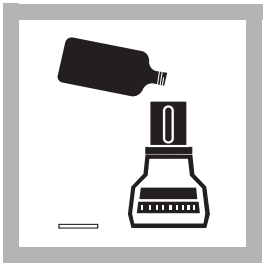
Note: *Preheat the COD Reactor to 150 °C for use later in the procedure.*



2. Press: **18 ENTER**

The display will show **mg/L, COD** and the **ZERO** icon**.**

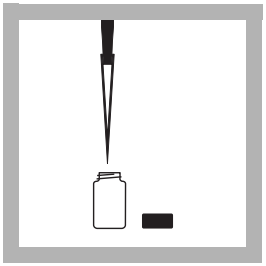
Note: *For alternate forms (O_2), press the* **CONC** *key.*



3. Homogenize 100 mL of sample for 30 seconds in a blender.

Note: *Blending promotes even distribution of solids and improves accuracy and reproducibility.*

Note: *Continue mixing the sample while pipetting if suspended solids are present.*



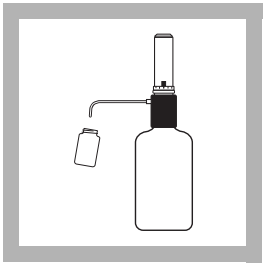
Chloride Removal Procedure

4. Using a TenSette Pipet or a pipet and safety bulb, pipet 9.0 mL of homogenized sample into an empty glass mixing cell. If the sample COD exceeds 1000 mg/L, dilute the sample as described in *Table 1*.

Note: *If suspended solids are present, continue mixing the sample while pipetting.*

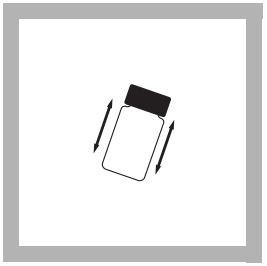
Caution: Some of the chemicals and apparatus used in this procedure may be hazardous to the health and safety of the user if inappropriately handled or accidentally misused. Please read all warnings and the safety section of this manual. Wear appropriate eye protection and appropriate clothing. If contact occurs, flush the affected area with running water. Follow all instructions carefully.

* U.S. Patent 5,556,787



5. Using an automatic dispenser or TenSette Pipet, add 1.0 mL of concentrated sulfuric acid to the mixing cell.

Note: *Mixing concentrated sulfuric acid and water is not additive. Adding 1.0 mL of concentrated sulfuric acid to 9.0 mL of sample does not result in a final volume of 10.0 mL. This factor is built into the calibration curve.*



6. Cap the cell tightly and invert it several times. The solution will become hot. Cool to room temperature before proceeding.

Note: *Acidified samples are stable for several months when refrigerated at 4 °C.*

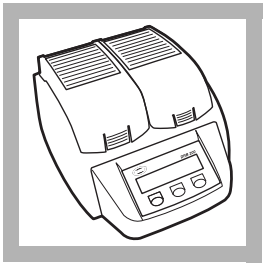
PREPARE BLANK

7. Prepare a blank (see note) by repeating Steps 4-6, substituting 9.0 mL of deionized water for the sample.

Note: *The reagent blank is stable and can be reused. Verify reagent blank quality by measuring the absorbance of the blank vs. a clean COD vial filled with deionized water. The absorbance range, when using chloride removal, should be about 1.31-1.36.*

Note: *Use a clean pipet or rinse it thoroughly.*

Note: *One blank must be run with each lot of reagents. Run all samples and blanks with the same lot of vials (lot number is on the container label).*



8. If not already on, turn on the DRB 200 Reactor and heat to 150 °C.

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*

Table 1 Dilution Table (for use with Chloride Removal Procedure Only)

Sample (mL)	Deionized Water (mL)	Range (mg/L COD)	Multiplication Factor
6.0	3.0	30-1500	1.5
3.0	6.0	60-3000	3
1.0	8.0	180-9000	9
0.5	8.5	360-18000	18

All dilutions require that the ratio of sample to sulfuric acid remain at 9:1. For other dilutions that are not listed in Table 1, simply add the sample volume + deionized water and divide by the sample volume to obtain the multiplication factor.

Example:

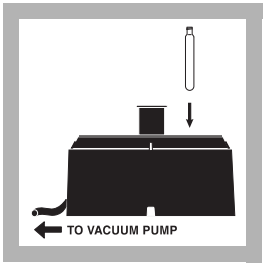
Dilute the sample to a range of 90-4500 mg/L COD

Sample Volume (2.0 mL) + Deionized water (7.0 mL) = Total Volume (9.0 mL)

$$\text{Multiplication Factor} = \frac{\text{Total Volume}}{\text{Sample Volume}} = \frac{9.0\ \text{mL}}{2.0\ \text{mL}} = 4.5$$

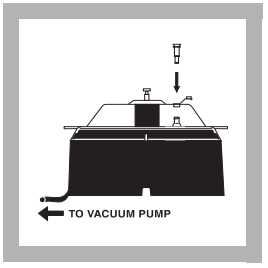
Standard test range is 20-1000 mg/L COD. Example Test Range = 4.5 (20) to 4.5 (1000) = 90-4500 mg/L COD

It is best to use 0.5 mL or more of sample for diluting. If sample values exceed 18,000 mg/L COD, use a separate sample dilution before the sample chloride removal procedure.

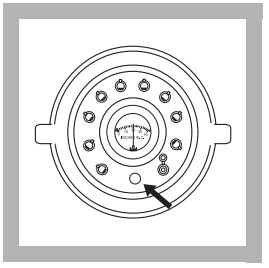


9. Label each Mn III COD vial and remove the cap. Place the vial in one of the numbered holes in the Vacuum Pretreatment Device (VPD)* base.

Note: *The VPD must be attached to a vacuum pump (not an aspirator-type vacuum) that can create a vacuum of 20 to 25 inches of mercury.*

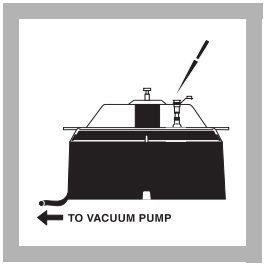


10. Place the VPD top on the base. Insert a fresh Chloride Removal Cartridge (CRC)** directly above each Mn III COD Reagent Vial. Plug any open holes in the VPD top using the stoppers provided.



11. Turn the vacuum pump on and adjust the vacuum regulator valve on top of the VPD until the internal gauge reads 20 inches of water.

Note: *The optimum setting allows the sample to flow through the CRC in about 30 to 45 seconds.*

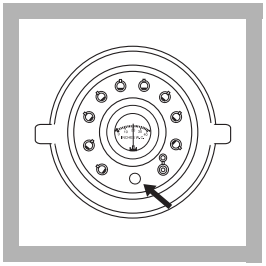


12. Pipet 0.60 mL of acidified sample (made in Steps 4-6) into the CRC. Pipet 0.60 mL of acidified blank into another CRC.

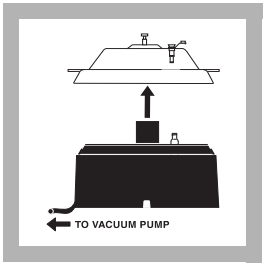
Note: *If the sample does not flow through the CRC, increase the vacuum until flow starts, then reduce the vacuum to 20 inches of water. Proceed as usual.*

* Patent Pending.

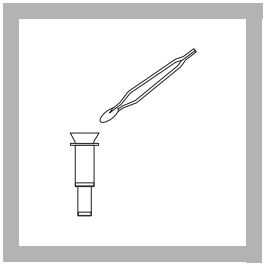
** U.S. patents 5,667,754 and 5,683,914.



13. Close the vacuum regulator valve completely to achieve full vacuum. After one minute under full vacuum, slide the VPD back and forth several times to dislodge any drops clinging to the cartridge.



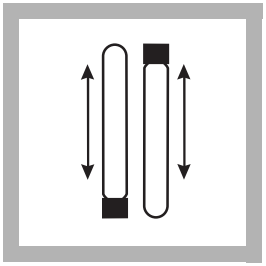
14. Open the VPD regulator valve to release the vacuum. Turn the pump off. Remove the VPD top and set it beside the base.



15. Use forceps to remove the filter from the top of each CRC. Place each filter in the corresponding Mn III COD Vial (use the numbers on the VPD as a guide).

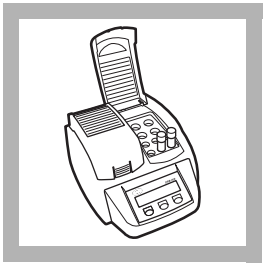
Note: *If the sample does not contain suspended solids, it is not necessary to transfer the filter to the digestion vial.*

Note: *To avoid cross contamination, clean forceps tips between samples by wiping with a clean towel or rinsing with deionized water.*



16. Remove the Mn III COD vial from the vacuum chamber and replace the original cap. Screw the cap on tightly. Invert several times to mix.

Note: *Dispose of the used Chloride Removal Cartridge. Do not reuse it.*

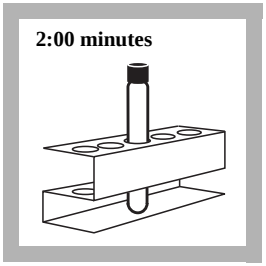


17. Place the vials in the DRB 200 Reactor that is preheated to 150 °C. Digest for 1 hour.

Note: *Boiling sample in the vials during digestion indicates the vial is not properly sealed; test results will be invalid.*

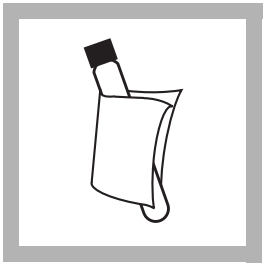
Note: *Samples can be digested up to 4 hours to oxidize more resistant organics. The prepared blank must be treated in the same manner.*

Note: *See DRB 200 user manual for selecting pre-programmed temperature applications.*



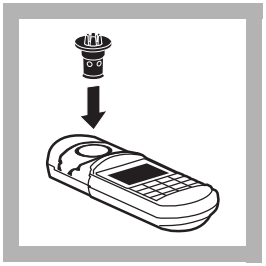
18. Remove the vials and place them in a cooling rack for two minutes to air cool. Then cool the vials to room temperature in a cool water bath or running tap water. This usually takes about three minutes.

Note: *Occasionally a vial will develop a colorless upper layer and a purple lower layer. Invert the vial several times to mix and proceed. This will not affect test results.*



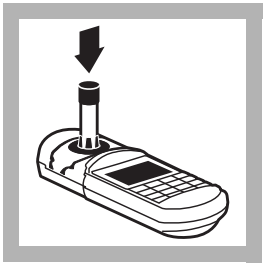
19. Remove the vials from the water and wipe with a clean, dry paper towel.

Invert the vials several times to mix.



20. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

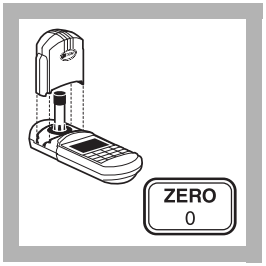
Note: *For increased performance, a diffuser band covers the light path holes on the adapter. Do not remove the diffuser band.*



21. Place the blank in the sample cell adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



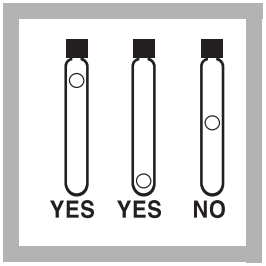
22. Tightly cover the sample cell with the instrument cap.

Note: *Clean the COD vial with a towel to remove fingerprints or other marks.*

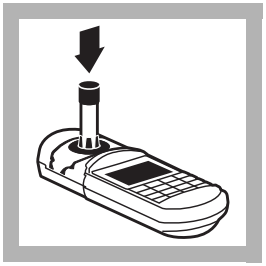
Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L COD



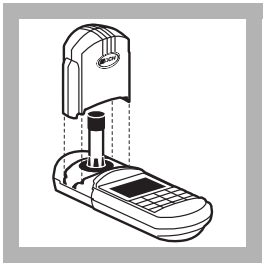
23. If the chloride removal was done, make sure the filter disc is not suspended in the middle of the vial; it can interfere with the instrument reading. Move it with gentle swirling or by lightly tapping the vial on the table top.



24. Place the sample in the adapter.

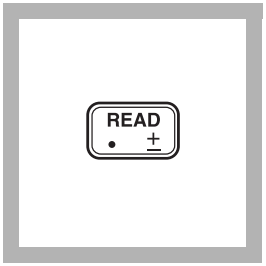
Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



25. Tightly cover the sample cell with the instrument cap.

Note: *Clean the COD vial with a towel to remove fingerprints or other marks.*



26. Press: **READ**

The cursor will move to the right, then the result in mg/L COD will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Note: *Adjust the result for any sample dilution.*

Sampling and Storage

Collect samples in clean glass bottles. Use plastic bottles only if they are known to be free of organic contamination. Test biologically active samples as soon as possible. Homogenize samples containing solids to assure representative samples. Samples treated with concentrated sulfuric acid to a pH of less than 2 (about 2 mL per liter) and refrigerated at 4 °C may be stored up to 28 days. Correct results for volume additions; see *Correcting for Volume Additions* (*Section 1*) for more information.

Accuracy Check

Standard Solution Method

Prepare an 800 mg/L COD standard solution by adding 0.6808 g of
dried (103 °C, overnight) potassium acid phthalate (KHP) to 1 liter of deionized water. Use 0.50 mL of this solution (0.60 mL for the chloride removal procedure) as the sample volume. The result should be
800 ±26 mg/L COD.

An 800 mg/L COD solution can also be purchased directly from Hach (see *Optional Reagents*).

Method Performance (for Manganic III COD without the chloride removal procedure)

Precision

In a single laboratory, using a standard solution of 800 mg/L COD and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±23 mg/L COD.

Estimated Detection Limit (EDL)

The EDL for program 18 is 14 mg/L COD. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Inorganic materials may also be oxidized by trivalent manganese and constitute a positive interference when present in significant amounts. Chloride is the most common interference and is removed by sample pretreatment with the Chloride Removal Cartridge. If chloride is known to be absent or present in insignificant levels, the pretreatment can be omitted. A simple way to determine if chloride will affect test results is to run

routine samples with and without the chloride removal, then compare results. Other inorganic interferences (i.e., nitrite, ferrous iron, sulfide) are not usually present in significant amounts. If necessary, these interferences can be corrected for after determining their concentrations with separate methods and adjusting the final COD test
results accordingly.

Ammonia nitrogen is known to interfere in the presence of chloride; it does not interfere if chloride is absent.

Summary of Method

Chemical oxygen demand (COD) is defined as "... a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant" (APHA Standard Methods, 19th ed., 1995). Trivalent manganese is a strong, non-carcinogenic chemical oxidant that changes quantitatively from purple to colorless when it reacts with organic matter. It typically oxidizes about 80% of the organic compounds. Studies have shown that the reactions are highly reproducible and test results correlate closely to Biochemical Oxygen Demand (BOD) values and hexavalent chromium COD tests. None of the oxygen demand tests provide 100% oxidation of all
organic compounds.

A calibration is provided which is based on the oxidation of Potassium Acid Phthalate (KHP). A different response may be seen in analyzing various wastewaters. The KHP calibration is adequate for most applications. The highest degree of accuracy is obtained when test results are correlated to a standard reference method such as BOD or one of the chromium COD methods. Special waste streams or classes will require a separate calibration to obtain a direct mg/L COD reading or to generate a correction factor for the precalibrated KHP response. The sample digestion time can be extended up to 4 hours for samples which are difficult to oxidize.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Chloride Removal Cartridges (CRC)	1	25/pkg	26618-25
Manganese III COD Reagent Vials, 20-1000 mg/L	1	25/pkg	26234-25
Sulfuric Acid, concentrated	1 mL	4 Kg	979-09
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Adapter, COD/TNT	1	each	48464-00
Blender, Osterizer, 120 Vac, 14-speed	1	each	26747-00
Blender Container, 118 mL	1	2/pkg	26748-00
Cap, with inert Teflon liner, for mixing bottle	varies	12/pkg	24018-12
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Forceps, extra fine point	1	each	26696-00
Mixing Bottle, glass, for sample + acid	1	each	24276-06
Pipet, TenSette, 1.0 to 10.0 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette	2	250/pkg	21997-25
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette	2	1000/pkg	21856-28
Test Tube Rack, stainless steel	1	each	18641-00
Vacuum Pretreatment Device (VPD)	1	each	49000-00
Vacuum Pump, 115 V	1	each	14697-00
Vacuum Pump, 230V	1	each	14697-02

OPTIONAL REAGENTS

COD Standard Solution, 800 mg/L COD	200 mL		26726-29
Oxygen Demand Standard (BOD, COD, TOC), 10-mL Ampules	16/pkg		28335-10
Potassium Acid Phthalate	500 g		315-34
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL		28332-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL		28331-49

OPTIONAL APPARATUS

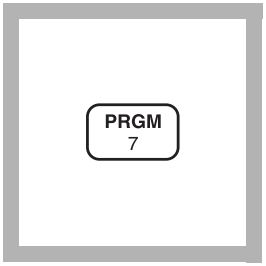
Dispenser for sulfuric acid		each	25631-37
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm			LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm			LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm			LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm			LTV082.52.30001

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

OXYGEN, DISSOLVED, High Range (0 to 15.0 mg/L O_2)

HRDO Method

For water and wastewater

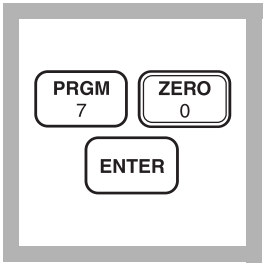


1. Enter the stored program number for dissolved oxygen, high range.

Press: **PRGM**

The display will show:

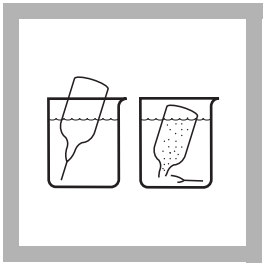
PRGM ?



2. Press: **70 ENTER**

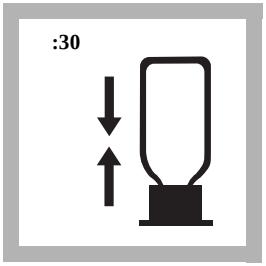
The display will show **mg/L, O2** and the **ZERO** icon.

3. Fill a sample cell (the blank) with at least 10 mL of sample. Fill a blue ampul cap with sample. Collect at least 40 mL of sample in a 50-mL beaker.



4. Fill a High Range Dissolved Oxygen AccuVac Ampul with sample.

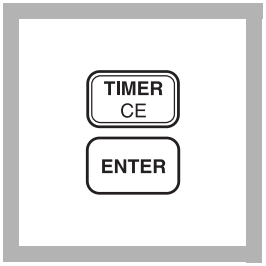
Note: *Keep the tip immersed while the ampul fills completely.*



5. Without inverting the ampul, immediately place the ampul cap that has been filled with sample securely over the tip of the ampul. Shake for about 30 seconds.

Note: *Accuracy is not affected by undissolved powder.*

Note: *The cap prevents contamination with atmospheric oxygen.*

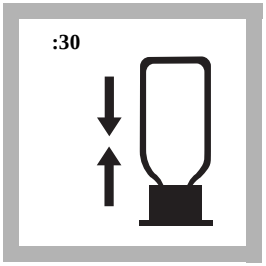


6. Press:

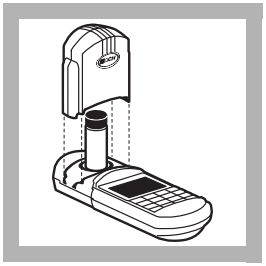
TIMER ENTER

A 2-minute reaction period will begin.

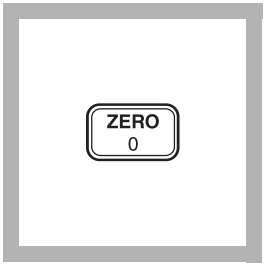
Note: *The two-minute period allows oxygen which was degassed during aspiration to redissolve in the sample and react.*



7. When the timer beeps, shake the ampul for 30 seconds.



8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

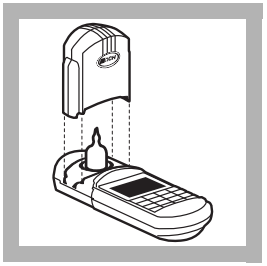


9. Press: **ZERO**

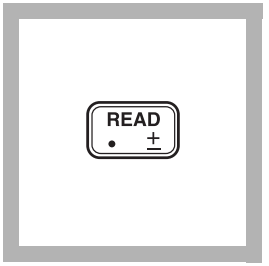
The cursor will move to the right, then the display will show:

0.0 mg/L O2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap. Wait approximately 30 seconds for the air bubbles to disperse from the light path.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L O_2 will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

The main consideration in sampling with the High Range Dissolved Oxygen AccuVac Ampul is to prevent the sample from becoming contaminated with atmospheric oxygen. This is accomplished by capping the ampul with an ampul cap in the interval between breaking open the ampul and reading the absorbance. If the ampul is securely capped, it should be safe from contamination for several hours. The absorbance will decrease by approximately 3% during the first hour and will not change significantly afterwards.

Sampling and sample handling are important considerations in obtaining meaningful results. The dissolved oxygen content of the water being tested can be expected to change with depth, turbulence, temperature, sludge deposits, light, microbial action, mixing, travel time and other factors. A single dissolved oxygen test rarely reflects the accurate over-all condition of a body of water. Several samples taken at different times, locations and depths are recommended for most reliable results. Samples must be tested immediately upon collection although only a small error results if the absorbance reading is taken several hours later.

Accuracy Check

The results of this procedure may be compared with the results of a dissolved oxygen meter (Cat. No. 51815-01).

Method Performance

Precision

In a single laboratory, using a standard solution of 8.0 mg/L O_2 determined by the Winkler method and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of
±0.41 mg/L O_2.

Estimated Detection Limit

The estimated detection limit for program 70 is 0.10 mg/L O_2. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Cr^{3+}	Greater than 10 mg/L
Cu^{2+}	Greater than 10 mg/L
Fe^{2+}	Greater than 10 mg/L
Mg^{2+}	Magnesium is commonly present in seawater and causes a negative interference. If the sample contains more than 50% seawater, the oxygen concentration obtained by this method will be 25% less than the true oxygen concentration. If the sample contains less than 50% seawater, the interference will be less than 5%.
Mn^{2+}	Greater than 10 mg/L
Ni^{2+}	Greater than 10 mg/L
NO_2^-	Greater than 10 mg/L

Summary of Method

The High Range Dissolved Oxygen AccuVac Ampul contains reagent vacuum sealed in a 12-mL ampul. When the AccuVac ampul is broken open in a sample containing dissolved oxygen, a yellow color forms, which turns purple as the oxygen reacts with the reagent. The color developed is proportional to the concentration of dissolved oxygen.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
High Range Dissolved Oxygen AccuVac Ampuls, with 2 reusable ampul caps	1 ampul	25/pkg	25150-25

REQUIRED APPARATUS

Beaker, 50 mL	1	each	500-41H
Caps, ampul, blue	varies	25/pkg	1731-25
Sample Cell, 10-20-25 mL, w/ cap	1	6/pkg	24019-06

OPTIONAL REAGENTS AND APPARATUS

AccuVac Dissolved Oxygen Sampler	each	24051-00
AccuVac Snapper Kit	each	24052-00
AccuVac Drainer	each	41036-00
BOD bottle and stopper, 300 mL	each	621-00
Dissolved Oxygen Meter, Portable HQ 10	each	51815-01
Dissolved Oxygen Reagent Set (Buret Method)	100 tests	23514-00
Dissolved Oxygen Reagent Set (Digital Titrator Method)	50 tests	22722-00

Dissolved oxygen may also be determined by titrimetric methods. Request Publication 8042 for additional information.

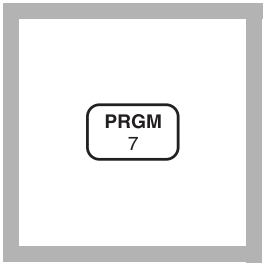
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

OXYGEN, DISSOLVED, Low Range (0 to 1000 µg/L O_2) For boiler feedwater

Indigo Carmine Method (Using AccuVac Ampuls)



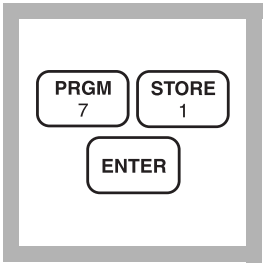
1. Enter the stored program number for low range dissolved oxygen (O_2).

Press: **PRGM**

The display will show:

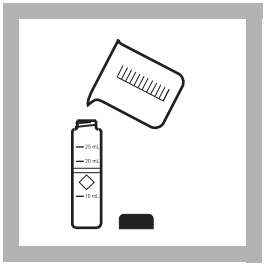
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



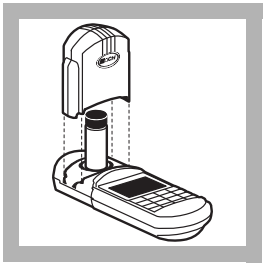
2. Press: **71 ENTER**

The display will show **µg/L, O2** and the **ZERO** icon**.**

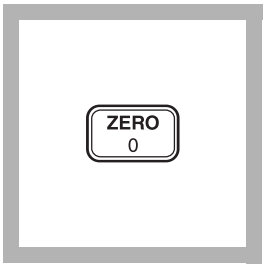


3. Fill a sample cell with at least 10 mL of sample (the blank).

Note: *Samples must be analyzed immediately and cannot be stored.*



4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

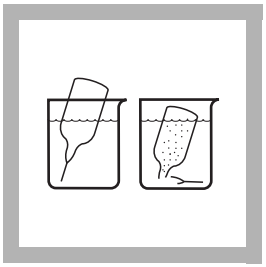


5. Press: **ZERO**

The cursor will move to the right, then the display will show:

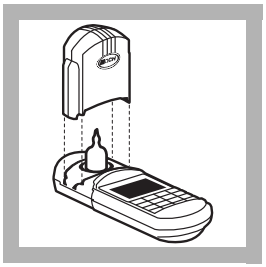
0 µg/L O2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



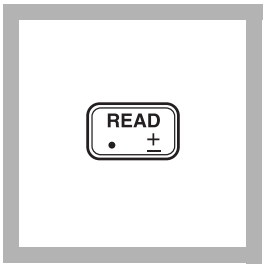
6. Collect at least 40 mL of sample in a 50-mL beaker. Fill a Low Range Dissolved Oxygen AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*



7. Immediately place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.

Note: *The ampuls will contain a small piece of wire to maintain reagent quality. The solution color will be yellow.*



8. Press: **READ**

The cursor will move to the right, then the result in µg/L dissolved oxygen will be displayed.

Note: *Use the initial reading. The reading is stable for 30 seconds. After 30 seconds, the ampul solution will absorb oxygen from the air.*

Sampling and Storage

The main consideration in this procedure is to prevent contaminating the sample with atmospheric oxygen. Sampling from a stream of water that is hard plumbed to the sample source is ideal. Use a funnel to maintain a continual flow of sample and yet collect enough sample to immerse the ampul. It is important not to introduce air in place of the sample. Rubber tubing, if used, will introduce unacceptable amounts of oxygen into the sample unless the length of tubing is minimized and the flow rate is maximized. Flush the sampling system with sample for at least 5 minutes.

Accuracy Check

The reagent blank for this test can be checked by following these steps:

a) Fill a 50-mL beaker with sample and add approximately 50 mg sodium hydrosulfite.

b) Immerse the tip of a Low Range Dissolved Oxygen AccuVac Ampul in the sample and break the tip. Keep the tip immersed while the ampul fills completely.

c) Determine the dissolved oxygen concentration according to the preceding procedure. The result should be 0 ±1 µg/L.

Method Performance

Precision

In a single laboratory, using a standard solution of 500 µg/L O_2 and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±2 µg/L O_2. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program #71 is 10 µg/L O_2. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Hydrazine	100,000 fold excess will begin to reduce the oxidized form of the indicator solution.
Sodium hydrosulfite	Reduces the oxidized form of the indicator solution and will cause a significant interference.

Excess amounts of sodium thioglycolate, sodium ascorbate, sodium ascorbate + sodium sulfite, sodium ascorbate + cupric sulfate, sodium nitrite, sodium sulfite, sodium thiosulfate, and hydroquinone do not cause significant interference.

Summary of Method

When the vacuum-sealed AccuVac ampul is broken open in a sample containing dissolved oxygen, the yellow reagent solution turns blue. The blue color is proportional to the dissolved oxygen concentration.

REQUIRED REAGENTS & APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Low Range Dissolved Oxygen AccuVac Ampuls	1 ampul	25/pkg	25010-25
Beaker, 50 mL	1	each	500-41H
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

OPTIONAL REAGENTS AND APPARATUS

AccuVac Snapper Kit	each	24052-00
Sodium Hydrosulfite, technical grade	500 g	294-34

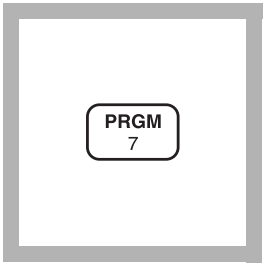
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

OZONE (0 to 0.25 mg/L O_3, 0 to 0.75 mg/L O_3 or 0 to 1.50 mg/L O_3) For water

Indigo Method (Using AccuVac Ampuls)

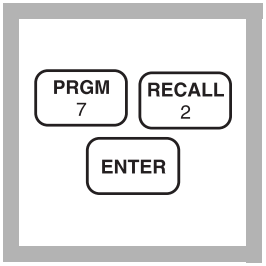


1. Enter the stored program number for Ozone (O_3) AccuVac ampuls.

Press: **PRGM**

The display will show:

PRGM ?

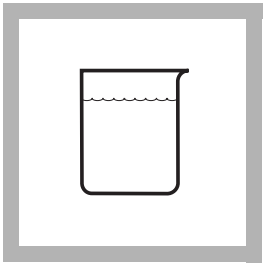


2. Press: **72 ENTER** for low range ozone

Press: **73 ENTER** for mid range ozone

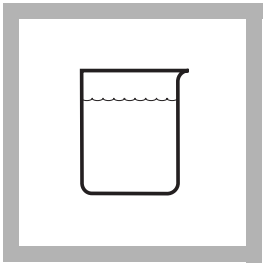
Press: **74 ENTER** for high range ozone.

The display will show **mg/L, O3** and the **ZERO** icon.



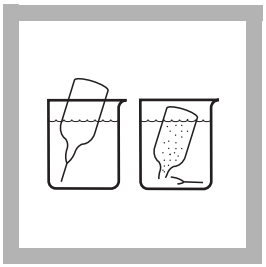
3. Gently collect at least 40 mL of sample in a 50-mL beaker.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis. See* Sampling and Storage *following these steps for proper collection.*



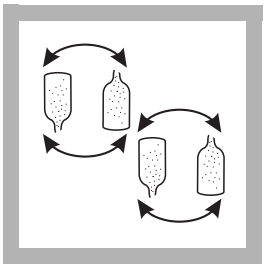
4. Collect at least 40 mL of ozone-free water (blank) in another 50-mL beaker.

Note: *Ozone-free water used for the blank may be deionized water or tap water.*



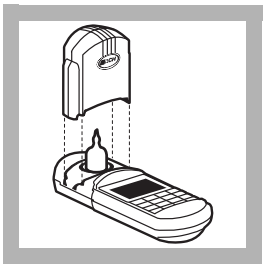
5. Fill one Indigo Ozone Reagent AccuVac Ampul with the sample and one ampul with the blank.

Note: *Keep the tip immersed while the ampul fills.*



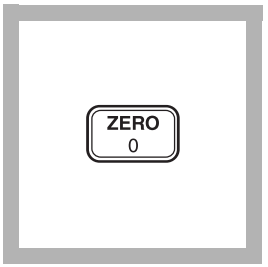
6. Quickly invert both ampuls several times to mix. Wipe off any liquid or fingerprints.

Note: *Part of the blue color will be bleached if ozone is present. (The sample will be lighter than the blank.)*



7. Place the **sample** AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.

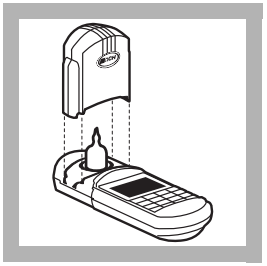
Note: *Standardization for this procedure is intentionally reversed.*



8. Press: **ZERO**

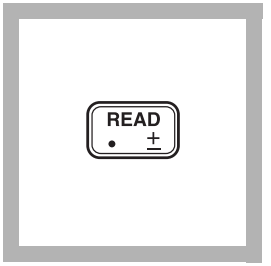
The cursor will move to the right, then the display will show:

0.00 mg/L O3



9. Place the AccuVac ampul containing the **blank** into the cell holder. Tightly cover the ampul with the instrument cap.

Note: *Standardization for this procedure is intentionally reversed.*



10. Press: **READ**

The cursor will move to the right, then the result in mg/L ozone will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling

The chief consideration when collecting a sample is to prevent the escape of ozone from the sample. The sample should be collected gently and analyzed immediately. Warming the sample or disturbing the sample by stirring or shaking will result in ozone loss. After collecting the sample, do not transfer it from one container to another unless absolutely necessary.

Stability of Indigo Reagent

Indigo is light-sensitive. Therefore, the AccuVac Ampuls should be kept in the dark at all times.

However, the indigo solution decomposes slowly under room light after filling with sample. The blank ampul can be used for multiple measurements during the same day.

Method Performance

Precision

In a single laboratory, using standard solutions of 0.15, 0.28 and 0.96 mg/L ozone for the low, mid and high range, respectively, and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.01, ±0.02 and ±0.02 mg/L O_3 for the low, mid and high range tests, respectively. For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for the programs #72, #73, and #74 is 0.02 mg/L O_3. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

The reagent formulation adjusts the sample pH to 2.5 after the ampul has filled. The indigo reagent reacts immediately and quantitatively with ozone. The blue color of indigo is bleached in proportion to the amount of ozone present in the sample. Other reagents in the formulation prevent chlorine interference. No transfer of sample is needed in the procedure. Therefore, ozone loss due to sampling is eliminated.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Ozone AccuVac Ampuls			
Select one or more based on range:			
0-0.25 mg/L	2 ampuls	25/pkg	25160-25
0-0.75 mg/L	2 ampuls	25/pkg	25170-25
0-1.50 mg/L	2 ampuls	25/pkg	25180-25
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Beaker, 50 mL	2	each	500-41H

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
AccuVac Ampule sampler	each	24051-00

For Technical Assistance, Price and Ordering

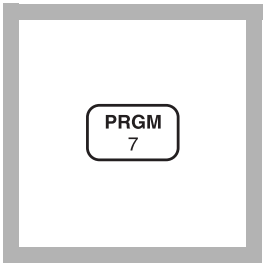
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

pH (6.5 to 8.5 pH units)

Colorimetric pH Determination Using Phenol Red

For water and wastewater

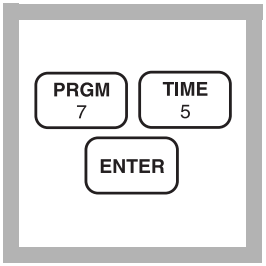


1. Enter the stored program number for the pH method.

Press: **PRGM**

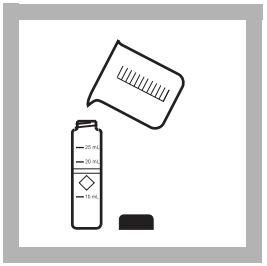
The display will show:

PRGM ?

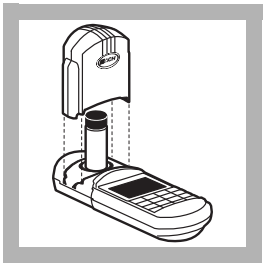


2. Press: **75 ENTER**

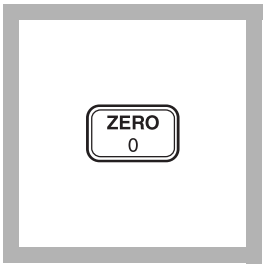
The display will show **PH** and the **ZERO** icon.



3. Fill a sample cell with 10 mL of sample (the blank).



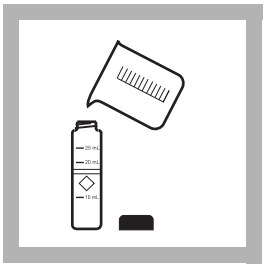
4. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

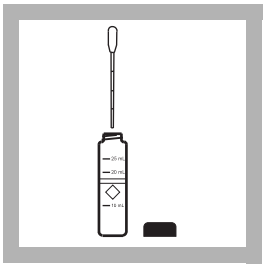
The cursor will move to the right, then the display will show:

6.0 PH

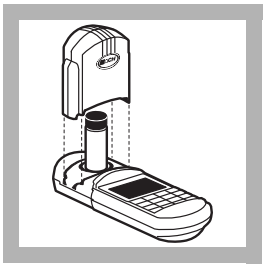


6. Fill another cell with 10 mL of sample.

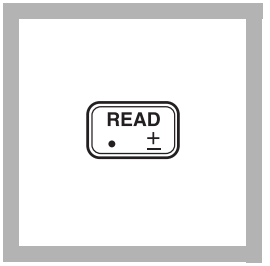
Note: *Sample temperature must be 21-29 °C.*



7. Using a disposable dropper, add 1 mL of Phenol Red Indicator Solution to the cell (the prepared sample). Cap the sample cell and invert twice to mix.



8. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



9. Press: **READ**

The cursor will move to the right, then the result in pH units will be displayed.

Note: *Use of the Standard Adjust feature is highly recommended. See* Accuracy Check.

Note: *Any reading below 6.5 pH units will be erroneous.*

Sampling and Storage

Analyze samples immediately for best results.

Accuracy Check

Standard Solution Method
Using a clear pH 7.0 buffer solution as the sample, perform the pH procedure as described above.

Method Performance

Precision
In a single laboratory using a standard solution of pH 7.0 and two lots of reagent with the instrument, a single operator obtained a standard deviation of less than 0.1 pH units.

Estimated Detection Limit
The estimated detection limit for program 75 is a pH of 6.5.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 7.0 buffer solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **7.0** to edit the standard concentration to match that of the standard used. See *Section 1, Standard Curve Adjustmen*t for more information. Press **ENTER** to complete the curve adjustment.

Interferences

Chlorine does not interfere at levels of 6 mg/L or lower.

Salt water (sea water) will interfere and cannot be analyzed using this method.

Summary of Method

This method uses a sulfonphthalein indicator (Phenol Red) to determine pH colorimetrically. Phenol Red has a working range of pH 6.8 (yellow) to 8.2 (red).

REQUIRED REAGENTS & APPARATUS

Description	Quantity Required Per Test	Units	Cat. No.
Dropper, 0.5 & 1.0 mL marks	1	20/pkg	21247-20
Phenol Red Indicator Solution, spec grade	1.0 mL	50 mL	26575-12
Sample Cells, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

pH 7.0 Buffer Solution		500 mL	12222-49

OPTIONAL APPARATUS

Description	Units	Cat. No.
Thermometer, -20 to 110 °C, Non-Mercury	each	26357-02

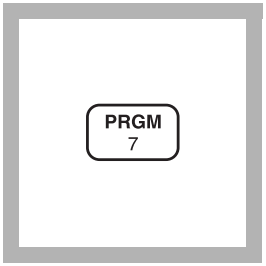
For Technical Assistance, Price and Ordering

In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHONATES (0-2.5 to 0-125 mg/L)

Persulfate UV Oxidation Method*



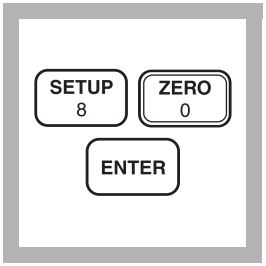
1. Enter the stored program number for phosphonates.

Press: **PRGM**

The display will show:

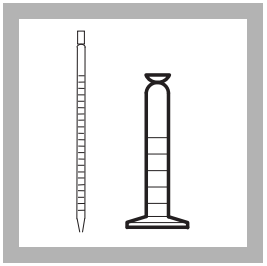
PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **80 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**



3. Choose the appropriate sample size from *Table 1* below. Pipet the chosen sample volume into a 50-mL graduated mixing cylinder. Dilute the sample to 50 mL with deionized water. Mix well.

Note: *Clean glassware with 1:1 hydrochloric acid, followed by a deionized water rinse. Do not use commercial detergents containing phosphates to clean glassware.*

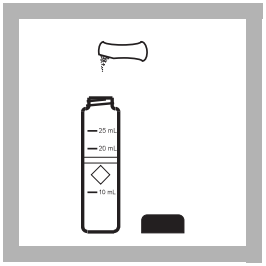
4. Fill a sample cell to the 10-mL mark with diluted sample from Step 3 (label this as the blank).

Fill another sample cell to the 25-mL mark with diluted sample from Step 3 (label this as the sample).

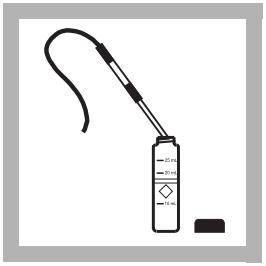
Table 1

Expected Range (mg/L phosphonate)	Sample Volume (mL)
0-2.5	50
0-5	25
0-12.5	10
0-25	5
0-125	1

* Adapted from Blystone, P.; Larson, P., *A Rapid Method for Analysis of Phosphonate Compounds*, International Water Conference, Pittsburgh, Pa. (Oct. 26-28, 1981).



5. Add the contents of one Potassium Persulfate for Phosphonate Powder Pillow to the cell labeled as "sample". Swirl to mix. This cell contains the prepared sample.

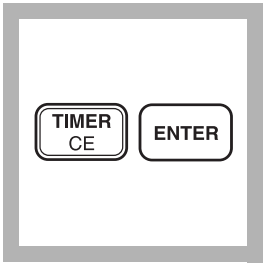


6. Insert the ultraviolet (UV) lamp into the prepared sample.

Note: *Wear UV safety goggles while the lamp is on.*

Note: *Do not handle the lamp surface. Fingerprints will etch the glass. Wipe lamp with a soft, clean tissue between samples. Do not use detergents with phosphates to wash glassware.*

Note: *A specially designed cord adapter is available for performing two digestions with a single power supply. A second UV lamp is required.*



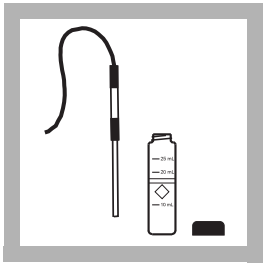
7. Turn on the UV lamp to digest the prepared sample.

Press: **TIMER ENTER**

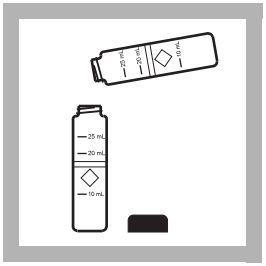
A 10-minute reaction period will begin.

Note: *Phosphonates are converted to ortho-phosphate in this step.*

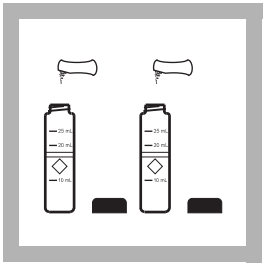
Note: *The digestion step may take less time. Contaminated samples or a weak lamp could result in incomplete digestion. Check efficiency by running a longer digestion to see if readings increase.*



8. When the timer beeps, turn off the UV lamp. Remove it from the sample cell.

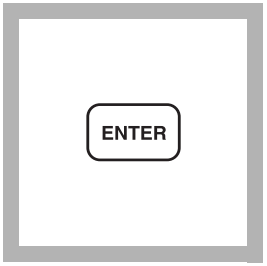


9. Pour 10 mL of sample from the cell labeled as "sample" into a second clean, dry sample cell. This is the prepared sample.



10. Add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow for 10-mL samples to each sample cell. Swirl immediately to mix.

Note: *A blue color will form if phosphate is present. Sample and blank cells may develop color.*

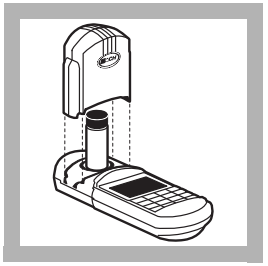


11. The display will show: **2:00 TIMER 2**

Press: **ENTER**

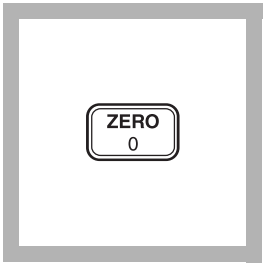
A two-minute reaction period will begin.

Note: *If sample is colder than 15 °C, 4 minutes are required for color development.*



12. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

Note: *Perform Steps 12-15 within three minutes after the timer beeps.*

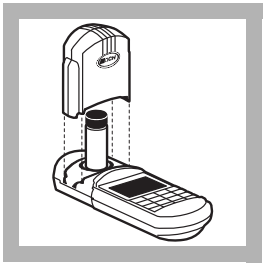


13. Press: **ZERO**

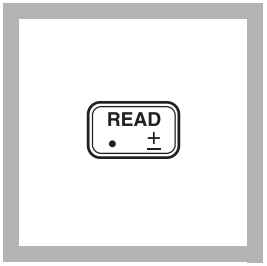
The cursor will move to the right, then the display will show:

0.0 mg/L PO4

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



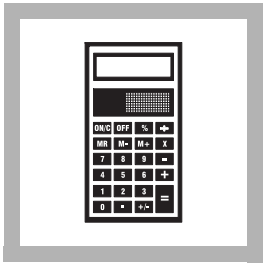
14. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



15. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate will be displayed. Multiply this value by the appropriate multiplier from *Table 2* to obtain the actual concentration of phosphonates as phosphate in the sample.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*



16. Results may be expressed in terms of a specific active phosphonate by using the appropriate conversion factor and the equation found in *Table 3*.

Table 2

Sample Volume (mL) (chosen in Step 3)	Multiplier
50	0.1
25	0.2
10	0.5
5	1.0
1	5.0
Phosphate concentration = Instrument Reading x Multiplier	

Table 3

Phosphonate Type	Conversion Factor
PBTC	2.84
NTP	1.050
HEDPA	1.085
EDTMPA	1.148
HMDTMPA	1.295
DETPMPA	1.207
HPA	1.49
Active Phosphonate (mg/L) = Phosphate concentration from Step 15 x Conversion Factor	

Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.
Do not use a commercial detergent. If prompt analysis is impossible, adjust the pH to 2 or less with about 2 mL of sulfuric acid, ACS, per liter of sample. Store at 4 °C (39 °F) or below. Preserved samples can be stored at least 24 hours. See *Section 1* for more information on dilution factors, cleaning instructions, etc.

Accuracy Check

Ideally, a solution containing a known amount of the phosphonate product being used should be prepared. This will check the UV conversion of phosphonate to orthophosphate.

Interferences

When testing a 5-mL sample volume, the following may interfere when present in concentrations exceeding those listed below:

The interference levels will decrease as the sample size increases. For example, copper does not interfere at or below 100 mg/L for a 5.00 mL sample. If the sample volume is increased to 10.00 mL, copper will begin to interfere above 50 mg/L.

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see *pH Interferences* in *Section 1*.

Phosphites and organophosphorus compounds other than phosphonates react quantitatively. Meta and polyphosphates do not interfere.

Interfering Substance	Level	Interfering Substance	Level
Aluminum	100 mg/L	EDTA	100 mg/L
Arsenate	all levels	Iron	200 mg/L
Benzotriazole	10 mg/L	Nitrate	200 mg/L
Bicarbonate	1000 mg/L	NTA	250 mg/L
Bromide	100 mg/L	Orthophosphate	15 mg/L
Calcium	5000 mg/L	Silica	500 mg/L
CDTA	100 mg/L	Silicate	100 mg/L
Chloride	5000 mg/L	Sulfate	2000 mg/L
Chromate	100 mg/L	Sulfide	All levels
Copper	100 mg/L	Sulfite	100 mg/L
Cyanide[1]	100 mg/L	Thiourea	10 mg/L
Diethanoldithiocarbamate	50 mg/L		

[1] Increase the UV digestion to 30 minutes.

Summary of Method

This method is directly applicable to boiler and cooling tower samples. The procedure is based on a UV catalyzed oxidation of phosphonate to orthophosphate. Range may be as low as 0 to 2.5 mg/L or as high
as 0 to 125 mg/L.

Phosphonate is converted to orthophosphate during the UV digestion. Both the sample and the blank will develop color if orthophosphate is present in the sample. The increase in color in the sample is proportional to the phosphate produced in the digestion.

REQUIRED REAGENTS

Phosphonates Reagent Set (100 tests) 24297-00
Includes: (2) 21060-69, (1) 20847-69

Description	Quantity Required Per Test	Unit	Cat. No
PhosVer 3 Phosphate Reagent Powder Pillows	2 pillows	100/pkg	21060-69
Potassium Persulfate Pillow for Phosphonate	1 pillow	100/pkg	20847-69
Water, deionized	varies	4 L	272-56

REQUIRED APPARATUS

Cylinder, mixing, graduated, 50 mL	1	each	1896-41
Goggles, UV safety	1	each	21134-00
Pipet, serological, 25 mL	1	each	2066-40
Pipet Filler, safety bulb	1	each	14651-00
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
UV Lamp with power supply, 115 V, with goggles	1	each	20828-00
OR			
UV Lamp with power supply, 230 V	1	each	20828-02

OPTIONAL REAGENTS

Hydrochloric Acid, 6.0 N (1:1)	500 mL		884-49
Sulfuric Acid, ACS	500 mL		979-49

OPTIONAL APPARATUS

pH Paper, 1 to 11 pH units	5 rolls/pkg		391-33
Pipet, serological, 2 mL	each		532-36
Pipet, TenSette, 1-10 mL	each		19700-10
Pipet Tips, for 19700-10 Tensette Pipet	50/pkg		21997-96
Thermometer, -20 to 110 °C, Non-Mercury	each		1877-01

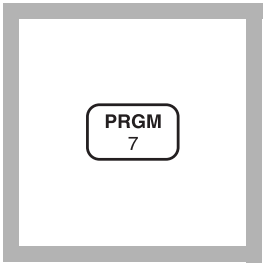
For Technical Assistance, Price and Ordering
In the U.S.A. call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, REACTIVE (0 to 2.50 mg/L PO_4^{3-}) For water, wastewater, seawater

(Also called Orthophosphate) PhosVer 3 (Ascorbic Acid) Method*

(Powder Pillows or AccuVac Ampuls) USEPA Accepted for wastewater analysis reporting**

Using Powder Pillows



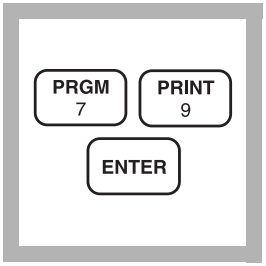
1. Enter the stored program number for reactive phosphorus, ascorbic acid method.

Press: **PRGM**

The display will show:

PRGM ?

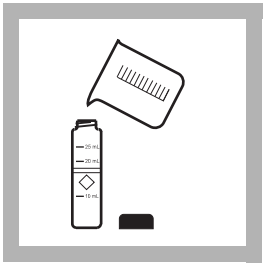
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **79 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

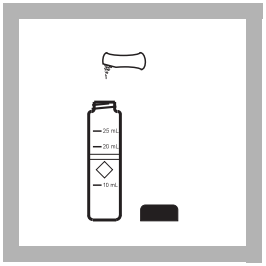
Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*



3. Fill a sample cell with 10 mL of sample.

Note: *For samples with extreme pH, see* Interferences *following these steps.*

Note: *Clean glassware with 1:1 HCl. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

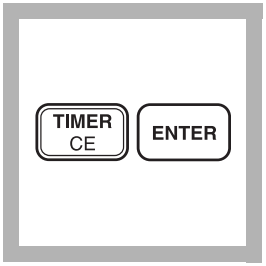


4. Add the contents of one PhosVer 3 Phosphate Powder Pillow for 10-mL sample to the cell (the prepared sample). Shake for 15 seconds.

Note: *A blue color will form if phosphate is present.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

** Procedure is equivalent to USEPA method 365.2 and Standard Method 4500-PE for wastewater.

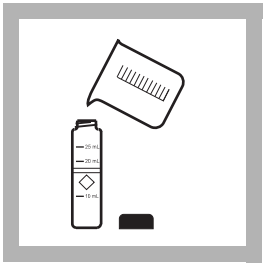


5. Press:

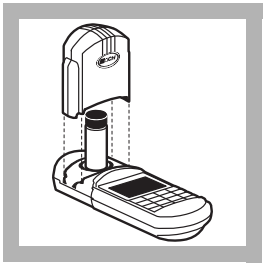
TIMER ENTER

A two-minute reaction period will begin. Perform Steps 6-8 during this period.

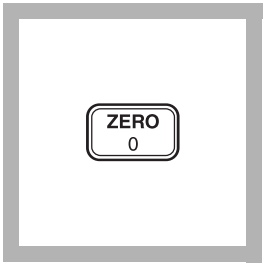
Note: *If the acid-persulfate digestion was used, an 8-10 minute reaction period is required.*



6. Fill another sample cell with 10 mL of sample (the blank).



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

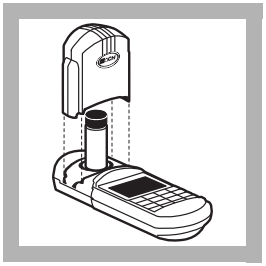


8. Press: **ZERO**

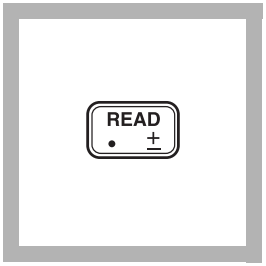
The cursor will move to the right, then the display will show:

0.00 mg/L PO4

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. After the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

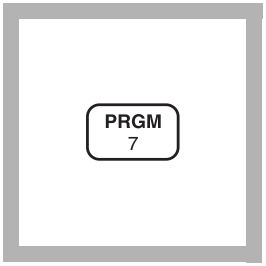


10. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *Standard Adjust may be performed using a 2.0-mg/L PO_4^{3-} standard; see Section 1.*

Using AccuVac Ampuls



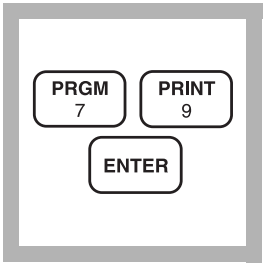
1. Enter the stored program number for reactive phosphorus-ascorbic acid method.

Press: **PRGM**

The display will show:

PRGM ?

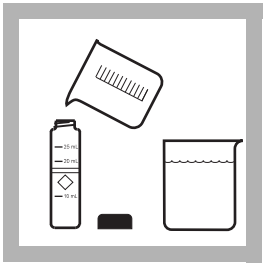
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **79 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

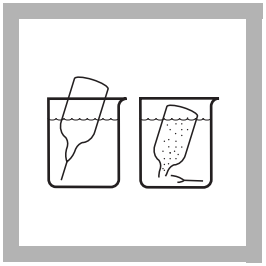
Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*



3. Fill a sample cell (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.

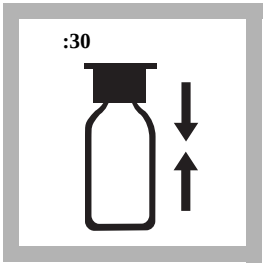
Note: *For samples with extreme pH, see* Interferences.

Note: *Clean glassware with 1:1 HCl. Rinse again with deionized water. Do not use detergent containing phosphates to clean glassware.*



4. Fill a PhosVer 3 Phosphate AccuVac Ampul with sample.

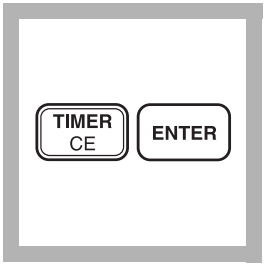
Note: *Keep the tip immersed while the ampul fills completely.*



5. Place an ampul cap securely over the tip of the ampul. Shake the ampul for about 30 seconds. Wipe off any liquid or fingerprints.

Note: *A blue color will form if phosphate is present.*

Note: *Accuracy is not affected by undissolved powder.*

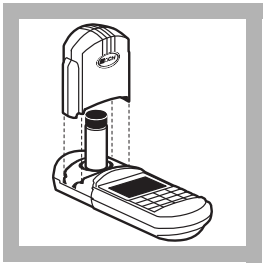


6. Press:

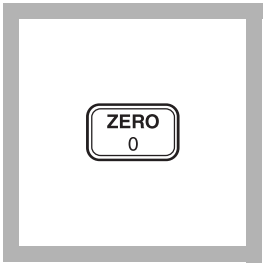
TIMER ENTER

A two-minute reaction period will begin. Perform Steps 7-8 during this period.

Note: *Use an 8-10 minute reaction period if determining total phosphorus following the acid-persulfate digestion.*



7. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

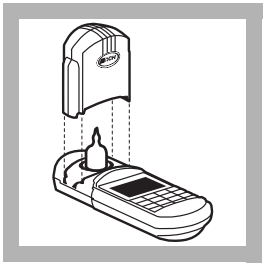


8. Press: **ZERO**

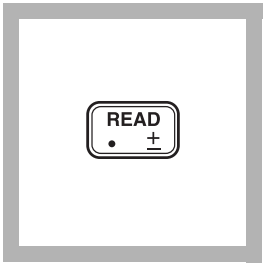
The cursor will move to the right, then the display will show:

0.00 mg/L PO4

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



9. After the timer beeps, place the AccuVac ampul into the cell holder. Tightly cover the ampul with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *Standard Adjust may be performed using a 2.0-mg/L PO_4^{3-} standard; see Section 1.*

Sampling and Storage

Collect sample in plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for up to 48 hours by filtering immediately and storing samples at 4 °C. Warm to room temperature before testing.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Phosphate PourRite Ampule Standard Solution, 50 mg/L as PO_4^{3-}.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper each and mix thoroughly.

d) For analysis with AccuVacs, transfer solutions to dry, clean 50 mL beakers to fill the AccuVac ampules. For analysis with powder pillows, transfer only 10 mL of solution to the sample cells.

e) Analyze each standard addition sample as described in the procedure. The phosphate concentration should increase 0.2 mg/L PO_4^{3-} for each 0.1 mL of standard added.

f) If these increases do not occur, see *Standard Additions* in *Section 1*.

Standard Solution Method

Prepare a 2.0 mg/L PO_4^{3-} standard solution by pipetting 4.0 mL of Phosphate Standard Solution, 50 mg/L as PO_4^{3-}, into an acid-washed Class A 100-mL volumetric flask. Dilute to volume with deionized water. Stopper and invert to mix. Use this solution in place of the sample in the procedure to insure the accuracy of the test. The mg/L PO_4^{3-} reading should be 2.00 mg/L.

Method Performance

Precision

In a single laboratory using a standard solution of 1.00 mg/L PO_4^{3-} and two lots of reagents with the instrument, a single operator obtained a standard deviation of ±0.05 mg/L PO_4^{3-}.

In a single laboratory using a standard solution of 1.00 mg/L PO_4^{3-} and two representative lots of AccuVac ampuls with the instrument, a single operator obtained a standard deviation of ±0.03 mg/L PO_4^{3-}.

Estimated Detection Limit (EDL)

The EDL for program 79 is 0.05 mg/L PO_4. For more information on the estimated detection limit, see *Section 1*.

Interference

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 200 mg/L
Arsenate	All levels
Chromium	Greater than 100 mg/L
Copper	Greater than 10 mg/L
Hydrogen sulfide	All levels
Iron	Greater than 100 mg/L
Nickel	Greater than 300 mg/L
Silica	Greater than 50 mg/L
Silicate	Greater than 10 mg/L
Turbidity or color	Large amounts may cause inconsistent results in the test because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles. For highly turbid or colored samples, add the contents of one Phosphate Pretreatment Pillow to 25 mL of sample. Mix well. Use this solution to zero the instrument.
Zinc	Greater than 80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment. pH 2 to 10 is recommended.

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a Phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows 10 mL sample size	1	Pillow 100/pkg	21060-69
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

REQUIRED REAGENTS & APPARATUS (Using AccuVac Ampuls)

PhosVer 3 Phosphate Reagent AccuVac Ampuls	1	ampul 25/pkg	25080-25
Beaker, 50 mL	1	each	500-41
Cap, ampul, blue	1	25/pkg	1731-25
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

OPTIONAL REAGENTS

Drinking Water Standard, Inorganic, F^-, NO_3^{-N}, PO_4^{3-}, SO_4^{2-}	500mL	28330-49
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phosphate Standard Solution, 1mg/L	500mL	2569-49
Phosphate Standard Solution, PourRite ampule, 50 mg/L as PO_4^{3-}, 2 mL	20/pkg	171-20
Phosphate Standard Solution, Voluette Ampul, 50 mg/L, 10 mL	16/pkg	171-10
Sodium Hydroxide Standard Solution, 5.0 N	100 mL* MDB	2450-32
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit for 10-ml ampules	each	21968-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, mixing, 25 mL, tall (3 required)	each	20886-40
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	100/pkg	13530-00
Flask, filtering, 500 mL	each	546-49
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *Sens**ion**™**1***, portable with electrode	each	51700-10
Pipet, 2 mL serological	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL TenSette Pipet	each	19700-01
Pipet Tips, for 19700-01	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet Filler, safety bulb	each	14651-00
Pipet, volumetric, Class A, 4.00 mL	each	14515-04
PourRite Ampule Breaker Kit	each	24846-00

Outside the U.S.A.—Contact the Hach office or distributor serving you.

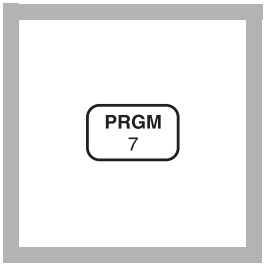
* Larger sizes available.

PHOSPHORUS, REACTIVE (0.00 to 5.00 mg/L PO_4^{3-})

PhosVer 3 Method, Test 'N Tube Procedure

For water, wastewater, and seawater

USEPA accepted for reporting wastewater analysis*



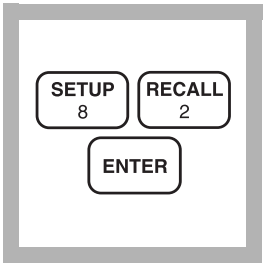
1. Enter the stored program number for reactive phosphorus (PO_4^{3-}), Test 'N Tube.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



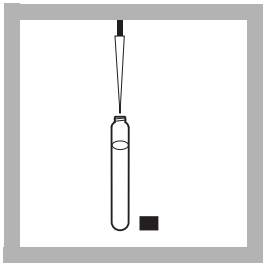
2. Press: **82 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*

3. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *A diffuser band covers the light path holes on the adapter to give increased performance.The band should NOT be removed.*



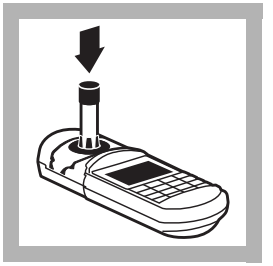
4. Use a TenSette Pipet to add 5.0 mL of sample to a Reactive Phosphorus Test 'N Tube Dilution Vial. Cap and mix.

Note: *For samples with extreme pH, see the* Interference *section.*

* Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P E for wastewater.

5. Clean the outside of the vial with a towel.

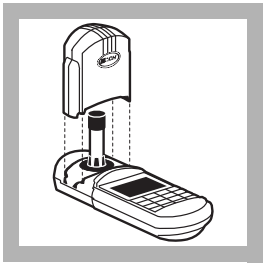
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



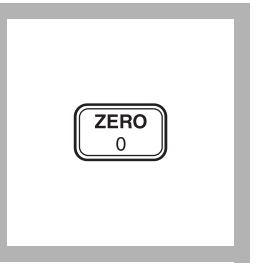
6. Place the sample vial into the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



7. Tightly cover the sample vial with the instrument cap.

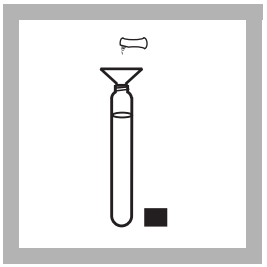


8. Press: **ZERO**

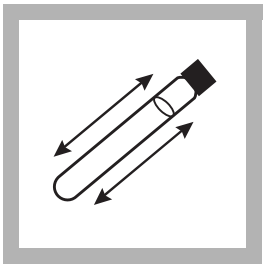
The cursor will move to the right, then the display will show:

0.00 mg/L PO4

Note: *For multiple samples, zero only on the first sample. Read the remaining samples after adding the PhosVer 3 Reagent.*

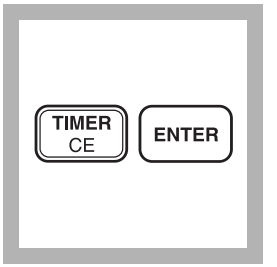


9. Using a funnel, add the contents of one PhosVer 3 Phosphate Powder Pillow to the vial.



10. Cap the vial tightly and shake for 10-15 seconds.

Note: *The powder will not completely dissolve.*



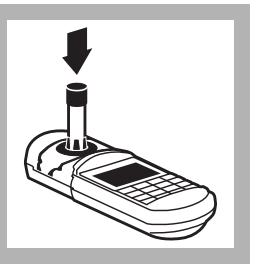
11. Press:

TIMER ENTER

A 2-minute reaction time will begin.

Note: *Read samples between 2 and 8 minutes after the addition of the PhosVer 3 reagent.*

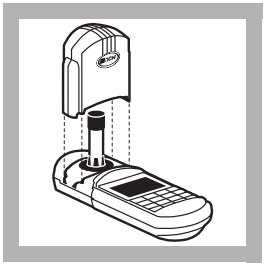
Note: *A blue color will develop if phosphate is present.*



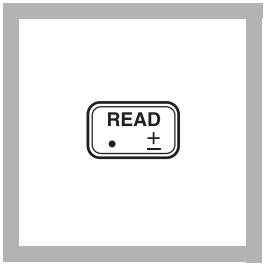
12. Immediately after the timer beeps, place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



13. Tightly cover the vial with the instrument cap.



14. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples for up to 48 hours by filtering immediately and storing at 4 °C. Warm to room temperature before analyzing the sample.

Accuracy Check

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as PO_4^{3-}.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.

d) Analyze each sample as described in the procedure; use 5.0 mL of the prepared standard additions for each test. The concentration should increase as follows: 0.2 mg/L, 0.4 mg/L, 0.6 mg/L PO_4^{3-}, respectively.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To check accuracy, use a 1.0 mg/L Phosphate Standard Solution listed under *Optional Reagents*. Or, prepare a 1.0-mg/L PO_4^{3-} standard by pipetting 2 mL of solution from a Phosphate Voluette Ampule Standard for Phosphate, 50 mg/L as PO_4^{3-}, into an acid-washed, Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

Method Performance

Precision

In a single laboratory, using a standard solution of 5.00 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.08 mg/L PO_4^{3-}.

Estimated Detection Limit (EDL)

The EDL for program 82 is 0.07 mg/L PO_4^{3-}. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding these listed below:

Substance	Interference Level and Treatment
Aluminum	200 mg/L
Arsenate	Interferes at any level
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Sulfide	6 mg/L. Sulfide interference may be removed by oxidation with Bromine Water as follows: 1. Measure 25 mL of sample into a 50-mL beaker. 2. Swirling constantly, add Bromine Water drop-wise until a permanent yellow color develops. 3. Swirling constantly, add Phenol Solution dropwise until the yellow color just disappears. Proceed with *step 1*.
Turbidity (large amounts)	May cause inconsistent results because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences (Section 1).

The PhosVer 3 Phosphate Reagent Powder Pillows should be stored in a cool, dry environment.

Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS

Reactive Phosphorus Test 'N Tube Reagent Set ..... 50 tests 27425-45
Includes: (1) 21060-46, (50) Orthophosphate Dilution Vials*

Description	Quantity Required Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows	1	50/pkg	21060-46
50 Orthophosphate Test 'N Tube Dilution Vials	1	50/pkg	*

REQUIRED APPARATUS

COD/TNT Adapter	1	each	48464-00
Funnel, micro	1	each	25843-35
Pipet, TenSette, 1 to 10 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	1	50/pkg	21997-96
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS

Bromine Water, 30 g/L	29 mL	2211-20
Drinking Water Standard, Inorganic, F^-, $NO_3^{-N,}$ PO_4^{3-}, SO_4^{2-}	500mL	28330-49
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phenol Solution, 30 g/L	29 mL	2112-20
Phosphate Standard Solution, 1 mg/L as PO_4^{3-}	500 mL	2569-49
Phosphate Standard Solution, Voluette ampule, 50 mg/L as PO_4^{3-}, 10 mL	16/pkg	171-10
Phosphate Standard Solution, PourRite ampule, 50 mg/L as PO_4^{3-}, 2 mL	20/pkg	171-20H
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Water, deionized	4 L	272-56

* These items are not sold separately.

OPTIONAL APPARATUS

Ampule Breaker, Pour Rite (2-mL ampule)	each	24846-00
Ampule Breaker Kit	each	21968-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, mixing, 25 mL (3 required)	each	20886-40
Dispenser, Repipet Jr., 2 mL	each	22307-01
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	100/pkg	13530-00
Flask, filtering, 500 mL	each	546-49
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable with electrode	each	51700-10
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01	50 pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet Filler, Safety Bulb	each	14651-00
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 2.00 mL	each	14515-36

For Technical Assistance, Price and Ordering

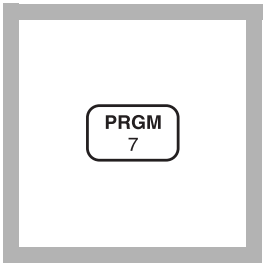
In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, REACTIVE (0 to 30.0 mg/L PO_4^{3-})

Amino Acid Method*

For water, wastewater, seawater



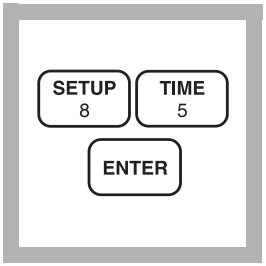
1. Enter the stored program number for reactive phosphate (PO_4^{3-}), amino acid method.

Press: **PRGM**

The display will show:

PRGM ?

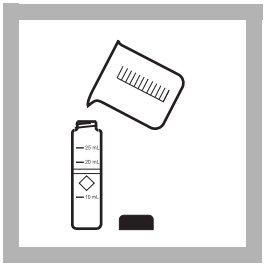
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



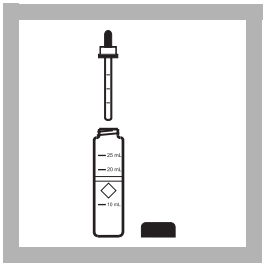
2. Press: **85 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon.

Note: *For alternate forms (P, P_2O_5), press* **CONC**.

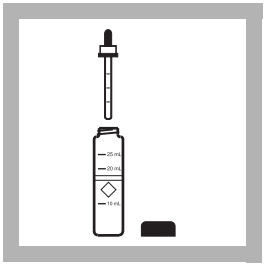


3. Fill a 25-mL sample cell with 25 mL of sample.



4. Add 1 mL of Molybdate Reagent using a 1-mL calibrated dropper.

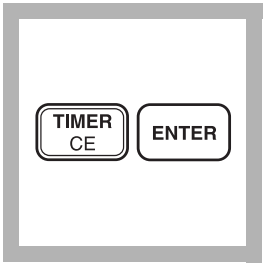
* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



5. Add 1 mL of Amino Acid Reagent Solution. Cap and invert several times to mix (the prepared sample).

Note: *A blue color will form if phosphate is present.*

Note: *You may substitute the contents of one Amino Acid Reagent Powder Pillow for 1 mL of Amino Acid Reagent Solution.*

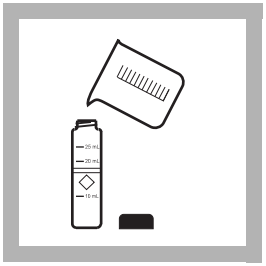


6. Press:

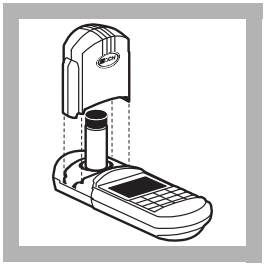
TIMER ENTER

A 10-minute reaction period will begin.

Note: *Perform Step 7 while the timer is running.*



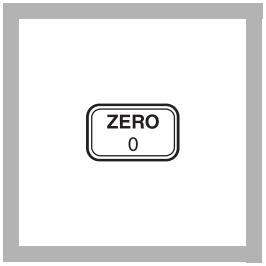
7. Pour 25 mL of sample (the blank) into a sample cell.



8. When the timer beeps, the display will show:

mg/L PO4

Place the blank into the cell holder. Cover the sample cell with the instrument cap.

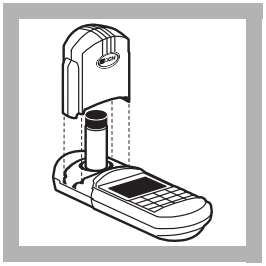


9. Press: **ZERO**

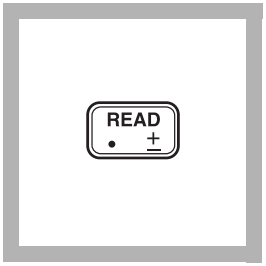
The cursor will move to the right, then the display will show:

0.0 mg/L PO4

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L PO_4 will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use a commercial detergent containing phosphate for cleaning glassware used in this test.

Analyze samples immediately for best results. If prompt analysis is not possible, preserve samples by filtering immediately and storing the sample at 4 °C (39 °F) for up to 48 hours.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Phosphate PourRite Ampule Standard Solution, 500 mg/L as PO_4^{3-}.

b) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 25-mL samples. Mix well.

c) Analyze each sample as described in the procedure. Each 0.1-mL addition of standard should cause an increase of 2.0 mg/L orthophosphate (PO_4^{3-}).

d) If these increases do not occur, see *Standard Additions* (*Section 1*) for more information.

Standard Solution Method

Prepare a 10.0-mg/L phosphate standard by pipetting 10.0 mL of a Phosphate Standard Solution, 50 mg/L as PO_4^{3-} into a 50-mL volumetric flask. Dilute to volume with deionized water.

Or, prepare a 10.0-mg/L PO_4^{3-} standard solution by using the TenSette Pipet to add 1.00 mL of Phosphate PourRite Ampule Standard,
500 mg/L as PO_4^{3-}, into a 50-mL volumetric flask. Dilute to volume
with deionized water.

Substitute this standard for the sample and perform the test as described. The mg/L PO_4^{3-} reading should be 10 mg/L.

Method Performance

Precision

In a single laboratory using a standard solution of 15.0 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.12 mg/L PO_4^{3-}.

Estimated Detection Limit

The estimated detection limit for program 85 is 0.14 mg/L PO_4^{3-}. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Calcium (Ca^{2+})	Greater than 10,000 mg/L as $CaCO_3$
Chloride	Greater than 150,000 mg/L as Cl^-
Colored samples	Add 1 mL of 10 N Sulfuric Acid Standard Solution to another 25-mL sample. Use this instead of untreated sample as the blank to zero the instrument. Use a pipet and pipet filler to measure the sulfuric acid standard.
High salt levels (Na^+)	May cause low results. To eliminate this interference, dilute the sample until two successive dilutions yield about the same result.
Magnesium	Greater than 40,000 mg/L as $CaCO_3$
Nitrites (NO_2^-)	Bleach the blue color. Remove nitrite interference by adding 0.05 g of sulfamic acid to the sample. Swirl to mix. Continue with Step 4.
Phosphates, high levels (PO_4^{3-})	As the concentration of phosphate increases, the color changes from blue to green, then to yellow and finally to brown. The brown color may suggest a concentration as high as 100,000 mg/L PO_4^{3-}. If a color other than blue is formed, dilute the sample and retest.
Sulfide (S^{2-})	Sulfide interferes. For samples with sulfide concentration less than 5 mg/L, sulfide interference may be removed by oxidation with Bromine Water as follows: **1.** Measure 50mL of sample into a 125-mL flask. **2.** Add Bromine Water dropwise with constant swirling until permanent yellow color develops. **3.** Add Phenol Solution dropwise until the yellow color just disappears. Use this sample in Steps 3 and 7.
Temperature	For best results, sample temperature should be 21 ±3 °C (70 ±5 °F).
Turbidity	May give inconsistent results for two reasons. Some suspended particles may dissolve because of the acid used in the test. Also, desorption of orthophosphate from particles may occur. For highly turbid samples, add 1 mL of 10 N Sulfuric Acid Standard Solution to another 25-mL sample. Use this instead of untreated sample as the blank to zero the instrument. Use a pipet and pipet filler to measure the sulfuric acid standard.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment.

Summary of Method

In a highly acidic solution, ammonium molybdate reacts with orthophosphate to form molybdophosphoric acid. This complex is then reduced by the amino acid reagent to yield an intensely colored molybdenum blue compound.

REQUIRED REAGENTS

	Cat. No.
High Range Reactive Phosphorus Reagent Set (100 Test)	22441-00

Includes: (1) 1934-32, (1) 2236-32

Description	Quantity Required Per Test	Units	Cat. No.
Amino Acid Reagent	1 mL	100 mL MDB*	1934-32
Molybdate Reagent	1 mL	100 mL MDB*	2236-32

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Description	Units	Cat. No.
Amino Acid Reagent Powder Pillow	100/pkg	804-99
Bromine Water, 30 g/L	29 mL	2211-20
Hydrochloric Acid Solution, 1:1 (6 N)	500 mL	884-49
Phenol Solution, 30 g/L	29 mL	2112-20
Phosphate Standard Solution, 50 mg/L PO_4^{3-}	500 mL	171-49
Phosphate Standard Solution, PourRite ampule, 500 mg/L PO_4^{3-}, 2 mL	20/pkg	14242-20
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfamic Acid	113 g	2344-14
Sulfuric Acid Standard Solution, 10 N	1 L	931-53
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49
Water, deionized	4 L	272-56

* Larger sizes available.

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Ampule Breaker Kit, PourRite	each	24846-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, 50 mL	each	508-41
Cylinder, graduated, mixing, 25 mL	each	20886-40
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	100/pkg	13530-00
Flask, filtering, 500 mL	each	546-49
Flask, erlenmeyer, 125 mL	each	505-43
Flask, volumetric, Class A, 50 mL	each	14574-41
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable with electrode	each	51700-10
Pipet, serological, 2.0 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Pipet Filler, safety bulb	each	12189-00
Spoon, measuring, 0.05 g	each	492-00
Thermometer, -20 to 110 °C, Non-Mercury	each	26357-02

For Technical Assistance, Price and Ordering

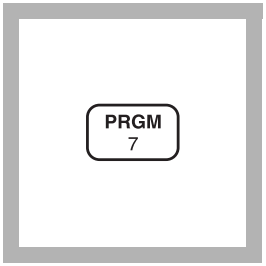
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, REACTIVE (0 to 45.0 mg/L PO_4^{3-}) For water and wastewater

(Also called Orthophosphate) Molybdovanadate Method*
(Reagent Solution or AccuVac Ampuls)

Using Reagent Solution

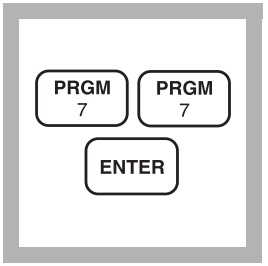


1. Enter the stored program number for high range phosphate (PO_4^{3-}) reagent solution.

Press: **PRGM**

The display will show:

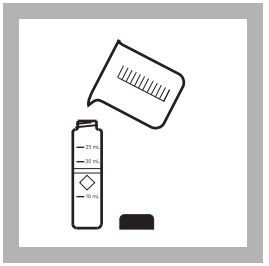
PRGM ?



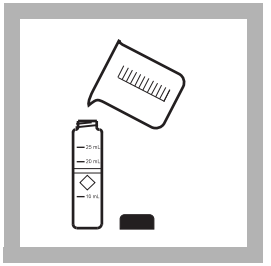
2. Press: **77 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*

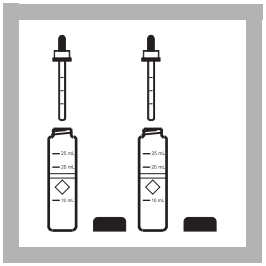


3. Fill a sample cell with 25 mL of deionized water (the blank).



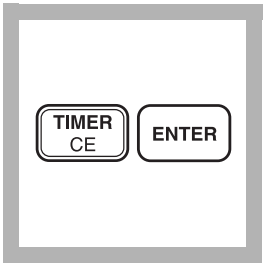
4. Fill another sample cell with 25 mL of sample (the prepared sample).

Note: *For best results, the sample temperature should be 20-25 °C.*



5. Add 1.0 mL of Molybdovanadate Reagent to each sample cell. Cap the cells and invert to mix.

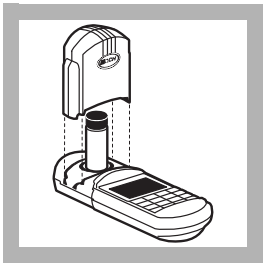
Note: *A yellow color will form if phosphate is present. A small amount of yellow will be present in the blank, because of the reagent.*



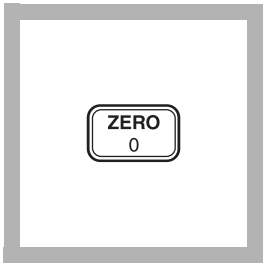
6. Press:

TIMER ENTER

A five-minute reaction period will begin.



7. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

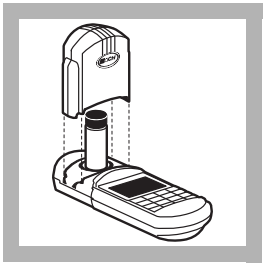


8. Press: **ZERO**

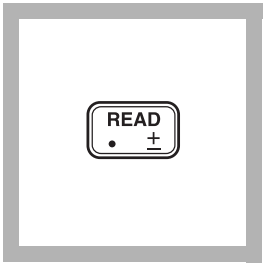
The cursor will move to the right, then the display will show:

0.0 mg/L PO4

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

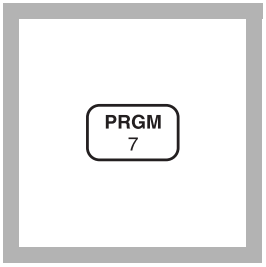


10. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (or alternate form) will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Using AccuVac Ampuls

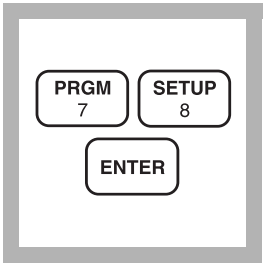


1. Enter the stored program number for high range phosphate (PO_4^{3-})-AccuVac Ampuls.

Press: **PRGM**

The display will show:

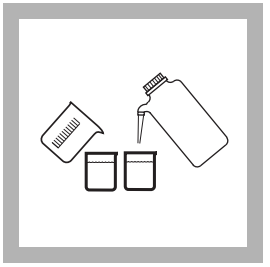
PRGM ?



2. Press: **78 ENTER**

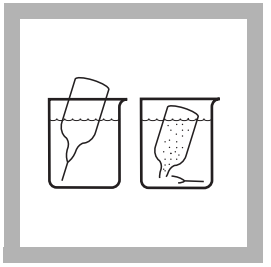
The display will show **mg/L, PO4** and the **ZERO** icon**.**

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*



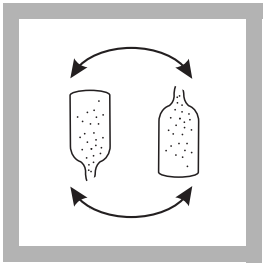
3. Collect at least 40 mL of sample in a 50-mL beaker. Pour at least 40 mL of deionized water into a second beaker.

Note: *For best results, sample temperature should be 20-25 °C.*



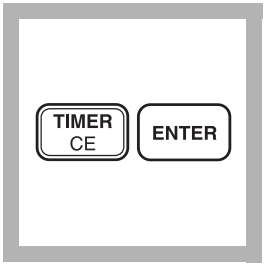
4. Fill a Molybdovanadate Reagent AccuVac Ampul with sample. Fill a second AccuVac Ampul with deionized water (the blank).

Note: *Keep the tip immersed while the ampul fills completely.*



5. Invert the ampul several times to mix, then wipe off any liquid or fingerprints.

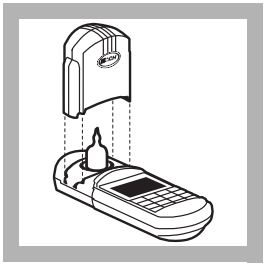
Note: *A yellow color will form if phosphate is present. A small amount of yellow will be present in the blank because of the reagent.*



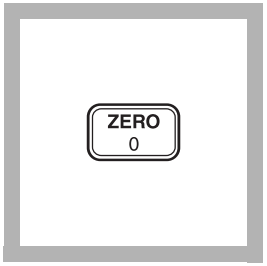
6. Press:

TIMER ENTER

A five-minute reaction period will begin.



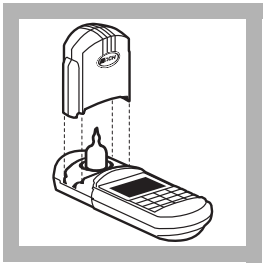
7. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



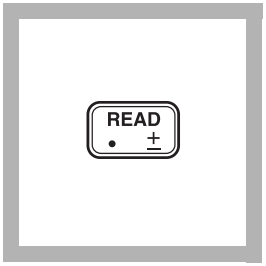
8. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L PO4



9. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



10. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (or alternate form) will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic or glass bottles that have been cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.

Do not use a commercial detergent containing phosphate for cleaning glassware used in this test.

Analyze samples immediately for best results. If prompt analysis is impossible, preserve samples by filtering immediately and storing at 4 °C for up to 48 hours.

Accuracy Check

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Phosphate Voluette Ampule Standard Solution, 500 mg/L as PO_4^{3-}.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper and invert to mix well.

d) For analysis with AccuVac Ampuls, transfer the spiked samples to clean, dry 50-mL beakers to facilitate filling of the ampuls. For analysis with reagent solution, transfer the spiked samples to
25-mL sample cells.

e) Analyze each sample as described in the procedure. Each 0.1-mL addition of standard should cause an increase of 2.0 mg/L PO_4^{3-}.

f) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

Standard Solution Method

Obtain a Hach Phosphate Standard Solution, 10.0 mg/L as phosphate. Using this solution as the sample, perform the phosphate procedure as described above.

Standard Adjust

To adjust the calibration curve using the reading obtained with the
10.0 mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustmen*t, *Section 1* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 30.0 mg/L PO_4^{3-}, two lots of reagent, and the instrument, a single operator obtained a standard deviation of ±0.1 mg/L PO_4^{3-} for the reagent solution method and a standard deviation of ±0.2 for the AccuVac Ampul method.

Estimated Detection Limit

The estimated detection limit for program 77 is 0.3 mg/L PO_4^{3-} and
0.4 mg/L PO_4^{3-} for program 78. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substances and Suggested Treatment

Interfering Substance	Interference Level and Treatment
Arsenate	Only interferes if sample is heated.
Iron, ferrous	Blue color caused by ferrous iron does not interfere if iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Only interferes if sample is heated.
Sulfide	Causes a negative interference. Remove interference as follows: **1.** Measure 50 mL of sample into an erlenmeyer flask. **2.** Add Bromine Water drop-wise with constant swirling until a permanent yellow color develops. **3.** Add Phenol Solution drop-wise until the yellow color just disappears. Proceed with step 4 of the procedure (step 3 if using the AccuVac procedure).
Extreme pH or highly buffered samples	May exceed buffering capacity of reagents. See Section 1, *pH Interferences*. Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause negative interference
The following do not interfere in concentrations up to 1000 mg/L: Pyrophosphate, tetraborate, selenate benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al^{3+}, Fe^{3+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+}, Li^{+}, Na^{+}, K^{+}, NH_4^{+}, Cd^{2+}, Mn^{2+}, NO_3^{-}, NO_2^{-}, SO_4^{2-}, SO_3^{2-}, Pb^{2+}, Hg^{+}, Hg^{2+}, Sn^{2+}, Cu^{2+}, Ni^{2+}, Ag^{+}, U^{4+}, Zr^{4+}, AsO_3^{-}, Br^{-}, CO_3^{2-}, $Cl0_4^{-}$, CN^{-}, IO_3^{-}, SiO_4^{4-}.	

Summary of Method

In the molybdovanadate method, orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid is formed. The intensity of the yellow color is proportional to the phosphate concentration.

REQUIRED REAGENTS AND APPARATUS (using Reagent Solution)

Description	Quantity Required Per Test		Units	Cat. No.
Molybdovanadate Reagent	2.0 mL	100 mL*	MDB	20760-32
Sample Cell, 10-20-25 mL, w/ cap	2		6/pkg	24019-06
Water, deionized	25 mL		4 L	272-56

REQUIRED REAGENTS AND APPARATUS (using AccuVac Ampuls)

Molybdovanadate Reagent AccuVac Ampuls	2		25/pkg	25250-25
Beaker, 50 mL	2		each	500-41H
Water, deionized	25 mL		4 L	272-56

OPTIONAL REAGENTS

Description	Units	Cat. No.
Bromine Water, 30 g/L	29 mL*	2211-20
Hydrochloric Acid Solution, 1:1 (6.0 N)	500 mL	884-49
Phenol Solution, 30 g/L	29 mL	2112-20
Phosphate Standard Solution, 10.0 mg/L as PO_4^{3-}	946 mL	14204-16
Phosphate Standard Solution, Voluette Ampule, 500 mg/L as PO_4^{3-}, 10 mL	16/pkg	14242-10
Sodium Hydroxide Standard Solution, 5.0 N	100 mL* MDB	2450-32
Sulfuric Acid, ACS	500 mL*	979-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49

OPTIONAL APPARATUS

AccuVac Snapper Kit	each		24052-00
Ampule Breaker Kit	each		21968-00
Cylinder, graduated, 25 mL	each		508-40
Cylinder, graduated, mixing, 25-mL	each		20886-40
Dispenser, fixed volume, 1.0 mL Repipet Jr.	each		21113-02
Flask, erlenmeyer, 50 mL	each		505-41
Flask, volumetric, Class A, 50 mL	each		14574-41
pH Paper, 1 to 11 pH units	5 rolls/pkg		391-33
pH Meter, *Sens**ion**™1*, portable with electrode	each		51700-10

* Contact Hach for larger sizes.

OPTIONAL APPARATUS (continued)

Description	Units	Cat. No.
Pipet, serological, 2.0 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Thermometer, -20 to 110 °C	each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, REACTIVE, HR (0.0 to 100.0 mg/L PO_4^{3-})

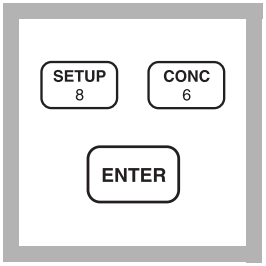
Molybdovanadate Method*, Test 'N Tube™ Procedure **For water and wastewater**

1. Enter the stored program number for phosphorus, reactive, high range, Test 'N Tube.

Press: **PRGM**

The display will show:

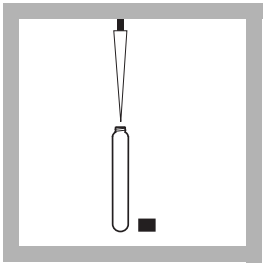
PRGM ?



2. Press: **86 ENTER**

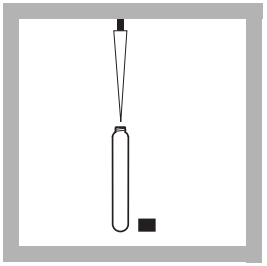
The display will show **mg/L, PO4** and the **ZERO** icon.

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*



3. Use a TenSette® Pipet to add 5.0 mL of deionized water to a Reactive High Range Phosphorus Test 'N Tube Vial (the blank).

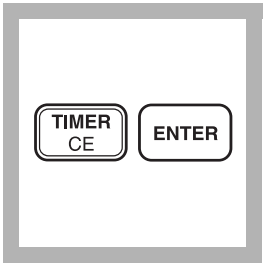
Cap and invert to mix.



4. Use a TenSette Pipet to add 5.0 mL of sample to a Reactive High Range Phosphorus Test 'N Tube Vial (the sample).

Cap and invert to mix.

Note: *For samples with extreme pH, see the Interference section.*



5. Press:

TIMER ENTER

A 7-minute reaction period will begin.

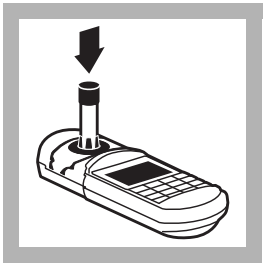
Note: *This reaction time is for samples at 23 °C (73 °F). If the sample temperature is 13 °C (55 °F), wait 15 minutes. If the sample temperature is 33 °C (91 °F), wait two minutes.*

6. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*

7. Clean the outside of the vials with a towel.

Note: *Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*

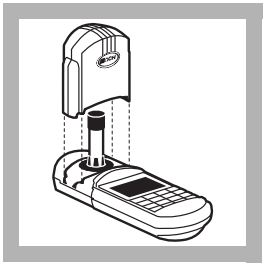


8. When the timer sounds, place the blank vial into the adapter.

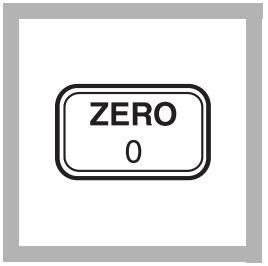
Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



9. Tightly cover the sample cell with the instrument cap.

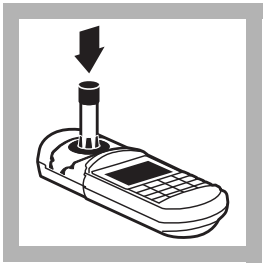


10. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L PO4

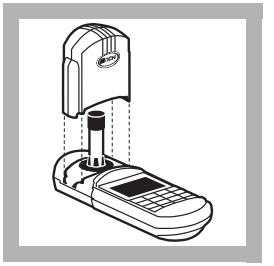
Note: *Reagent blanks for each lot of reagent may be used more than once. At room temperature, the reagent blank is stable for as long as three weeks; then prepare a new one.*



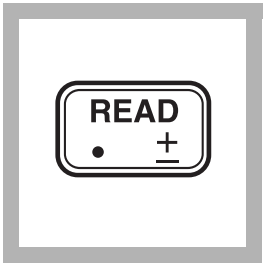
11. Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



12. Tightly cover the vial with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *For best results, use Standard Adjust with each new lot of reagent. (See* Accuracy Check.*)*

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphate for cleaning the glassware used in this test.

For best results, analyze the samples immediately after collection. If prompt analysis is impossible, preserve the samples for up to 748 hours by filtering immediately and storing at 4 °C. The sample should have a neutral (6–8) pH and be at room temperature before analysis.

Accuracy Check

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

Standard Additions Method

a. Fill three 10-mL graduated mixing cylinders with 10 mL of sample.

b. Snap the neck off a Voluette™ Ampule of Phosphate Standard Solution, 500 mg/L as PO_4^{3-} (Cat. No. 14242-10).

c. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 10-mL aliquots of sample prepared in *step a*. Mix well.

d. Analyze each sample from *step c* as described in the procedure; use 5.0 mL of the prepared sample for each test. The concentration should increase as follows: 5 mg/L, 10 mg/L, and 15 mg/L PO_4^{3-}, respectively.

e. If these increases do not occur, see *Standard Additions* in *Section 1* of the *DR/890 Procedures Manual* for more information.

Standard Solution Method

To check accuracy, prepare an 80 mg/L PO_4^{3-} standard by pipetting 8.0 mL of solution from a 10-mL Voluette Ampule of Phosphate Standard Solution, 500 mg/L as PO_4^{3-}, into an acid-cleaned 50-mL Class A volumetric flask. Fill to the line with deionized water. Substitute this standard for the sample and perform the procedure as described.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 80 mg/mL PO_4^{3-} standard solution, press the **SETUP** key and

scroll, using the arrow keys, to the **STO** option. Press **ENTER** to activate the standard adjust option. Then enter 80.0 to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1* of the *Procedures Manual* for more information.

Interferences

Large amounts of sample turbidity may cause inconsistent results in the test because the acid present in the reagents may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.

The following may interfere when present in concentrations exceeding these listed below:

Substance	Interference Level and Treatment
Arsenate	Causes positive interference if the sample is heated.[1]
Iron, ferrous	Blue color caused by ferrous iron does not interfere if the iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Causes positive interference if the sample is heated.*
Sulfide	Causes a negative interference. Remove interference as follows: **1.** Measure 50 mL of sample into an Erlenmeyer flask. **2.** Add Bromine Water drop-wise with constant swirling until a permanent yellow color develops. **3.** Add Phenol Solution drop-wise until the yellow color just disappears. Proceed with *step 1* of the procedure.
Extreme pH or highly buffered samples	May exceed buffering capacity of the reagents. See *pH Interferences* in *Section 1* of the *DR/890 Procedure Manual*. Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause a negative interference.
Temperature, cold (less than 20 °C)	Causes a negative interference.
Temperature, hot (greater than 25 °C)	Causes a positive interference.
The following do not interfere in concentrations up to 1000 mg/L: Pyrophosphate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al^{3+}, Fe^{3+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+}, Li^{+}, Na^{+}, K^{+}, NH_4^{+}, Cd^{2+}, Mn^{2+}, NO_3^{-}, NO_2^{-}, SO_4^{2-}, SO_3^{2-}, Pb^{2+}, Hg^{+}, Hg^{2+}, Sn^{2+}, Cu^{2+}, Ni^{2+}, Ag^{+}, U^{4+}, Zr^{4+}, AsO_3^{-}, Br^{-}, CO_3^{2-}, ClO_4^{-}, CN^{-}, IO_3^{-}, SiO_4^{4-}.	

[1] Gentle warming of the sample to reach room temperature will not cause this substance to interfere.

Method Performance

Precision

In a single laboratory, using a standard solution of 80.0 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±3.0 mg/L PO_4^{3-}.

Estimated Detection Limit (EDL)

The EDL for program 86 is 7.0 mg/L PO_4^{3-}. For more information on derivation and use of Hach's estimated detection limit, see *Section 1* of the *DR/890 Procedures Manual*.

Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Consult the Material Safety Data Sheet for information specific to the reagent used.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet for information specific to the reagents used.

Summary of Method

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration.

Installing this Program on the DR/800

This procedure will add the current method as a new Hach program to your DR/800.

1. Turn the DR/800 on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key two times so that the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.

6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change any number you have already entered.

Line Number	Entry	Line Number	Entry
1	86	29	0
2	4	30	80
3	73	31	50
4	0	32	79
5	0	33	53
6	0	34	0
7	0	35	62
8	65	36	166
9	56	37	246
10	217	38	148
11	21	39	63
12	66	40	63
13	157	41	78
14	197	42	252
15	30	43	4
16	0	44	76
17	0	45	128
18	0	46	0
19	0	47	15
20	80	48	1
21	79	49	164
22	52	50	0
23	0	51	0
24	0	52	0
25	80	53	0
26	0	54	80
27	0	55	0
28	0	56	255

REQUIRED REAGENTS

High Range Reactive Phosphorus Test 'N Tube™ Reagent Set.....50 vials.....27673-45
Includes: (50) Reactive High Range Phosphorus Test 'N Tube™ Vials*, (2) 272-42

Description	Quantity Required Per Test	Unit	Cat. No.
Reactive High Range Phosphorus Test 'N Tube™ Vials	1	50/pkg	*
Water, deionized		100 mL	272-42

REQUIRED APPARATUS

COD/TNT Adapter for DR/800 Series	1	each	48464-00
Pipet, TenSette®, 1 to 10 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette® Pipet	1	50/pkg	21997-96
Test Tube Rack	1–3	each	18641-00

OPTIONAL REAGENTS

Bromine Water, 30 g/L	29 mL**	2211-20
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phenol Solution, 30 g/L	29 mL	2112-20
Phosphate Standard Solution, PourRite ampule, 500 mg/L as PO_4^{3-}, 2 mL	20/pkg	14242-20
Phosphate Standard Solution, Voluette ampule, 500 mg/L as PO_4^{3-}, 10 mL	16/pkg	14242-10
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49

OPTIONAL APPARATUS

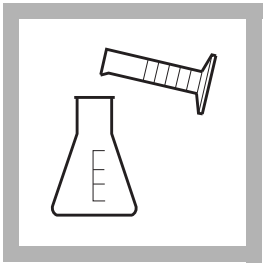
Ampule Breaker Kit	each	21968-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, mixing, 10 mL, 3 required	each	20886-38
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	200/pkg	13530-00
Flask, filtering, 500 mL	each	546-49
Flask, volumetric, Class A, 50-mL	each	14574-41
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion**™1*, portable with electrode	each	51700-10
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette® Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette® Pipet	1000/pkg	21856-28
Pipet Tips, for 19700-10 TenSette® Pipet	250/pkg	21997-25
Pipet, volumetric, Class A, 5.00-mL	each	14515-37
Pipet, volumetric, Class A, 8.00-mL	each	14515-08
PourRite™ Ampule Breaker	each	24846-00

* These items are not sold separately.
** Larger sizes available.

PHOSPHORUS, ACID HYDROLYZABLE

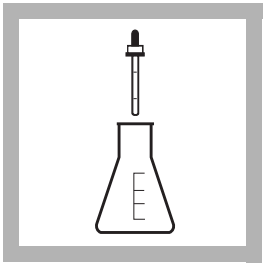
Hydrolysis to Orthophosphate Method*

For water, wastewater, seawater



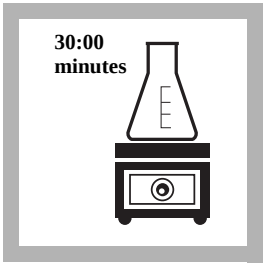
1. Measure 25 mL of sample into a 50-mL erlenmeyer flask using a graduated cylinder.

Note: *Wash all glassware with 6 N hydrochloric acid. Rinse with deionized water. Do not use detergents containing phosphate to clean glassware.*



2. Add 2.0 mL of 5.25 N Sulfuric Acid Solution.

Note: *Use the 1-mL calibrated dropper provided.*

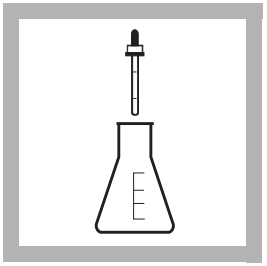


3. Place the flask (the prepared sample) on a hot plate. Boil gently for 30 minutes.

Note: *Samples should be concentrated to less than 20 mL for best recovery. After concentration, maintain the volume near 20 mL by adding small amounts of deionized water. Do not exceed 20 mL.*

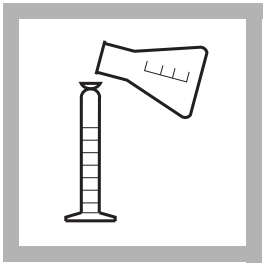
4. Cool the hot prepared sample to room temperature.

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



5. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution to the prepared sample. Swirl to mix.

Note: *Use the 1-mL calibrated dropper provided.*



6. Pour the prepared sample into a graduated cylinder. Add deionized water rinsings from the flask to return the volume to 25 mL. Proceed with the appropriate reactive phosphorus test.

Note: *Results of the reactive phosphorus test at this point will include the orthophosphate plus the acid-hydrolyzable (condensed) phosphate. The condensed phosphate concentration is determined by subtracting the results of a reactive phosphorus test on an untreated sample from this result. Make sure both results are in the same chemical form and units.*

Sampling and Storage

Analyze samples immediately after collection for best results. If prompt analysis is not possible, samples may be preserved up to 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

Interferences

If the sample is turbid, use 50 mL of sample and double the reagent volumes. Use the hydrolyzed sample to zero the instrument in the reactive phosphorus procedure. This compensates for any turbidity dissolved by this procedure.

Summary of Method

This procedure lists the necessary steps to convert condensed phosphate forms (meta-, pyro- or other polyphosphates) to reactive orthophosphate before analysis. The procedure uses acid and heat to hydrolyze the sample. Organic phosphates are not converted to orthophosphate by this process, but a very small fraction may be unavoidably included in the result. Thus, the "acid hydrolyzable" phosphate results are primarily a measure of inorganic phosphorus. This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorous content of the sample.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Drinking Water Standard, Inorganic, F^-, $NO_3^{-N,}$ PO_4^{3-}, SO_4^{2-}	500 mL		28330-49
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL* MDB	2450-32
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL* MDB	2449-32
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL		28332-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL		28331-49

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	2	each	508-40
Flask, erlenmeyer, 50 mL	1	each	505-41

OPTIONAL REAGENTS

Hydrochloric Acid, 6 N	500 mL		884-49
Water, deionized	4 L		272-56

OPTIONAL APPARATUS

Cylinder, graduated, 50 mL		each	508-41
Flask, erlenmeyer, 125 mL		each	505-43
Hot Plate, 4" diameter, 120 Vac		each	12067-01
Hot Plate, 4" diameter, 240 Vac		each	12067-02
Pad, cooling, 4" x 4"		each	18376-00
pH indicator Paper, 1 to 11 pH	5	rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable with electrode		each	51700-10
Thermometer, -20 to 110 °C, Non-Mercury		each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

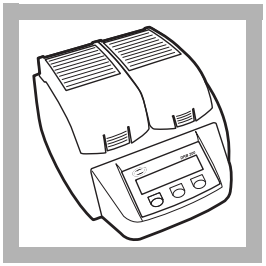
Outside the U.S.A.—Contact the Hach office or distributor serving you.

* Contact Hach for larger sizes.

PHOSPHORUS, ACID HYDROLYZABLE (0.00 to 5.00 mg/L PO_4^{3-})

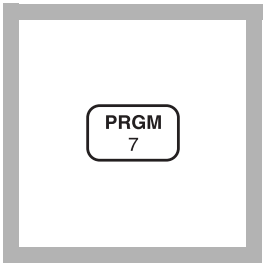
PhosVer 3 with Acid Hydrolysis Test 'N Tube™ Procedure

For water, wastewater, and seawater



1. Turn on the COD Reactor. Heat to 150 °C.

Note: *See DRB200 instrument manual for selecting preprogrammed temperature applications.*



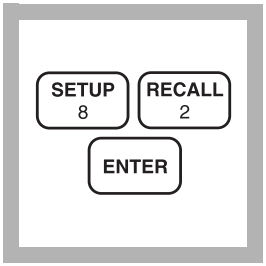
2. Enter the stored program number for acid hydrolyzable phosphorus (PO_4^{3-}), Test 'N Tube.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



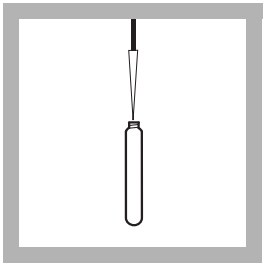
3. Press: **82 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

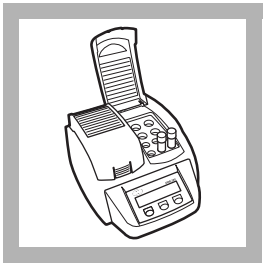
Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*

4. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

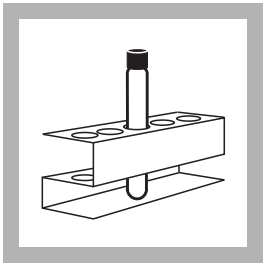
Note: *A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*



5. Use a TenSette Pipet to add 5.0 mL of sample to a Total and Acid Hydrolyzable Test Vial. Cap and mix.

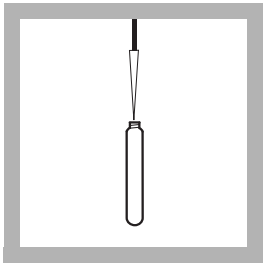


6. Heat the vial in the DRB200 Reactor for 30 minutes.

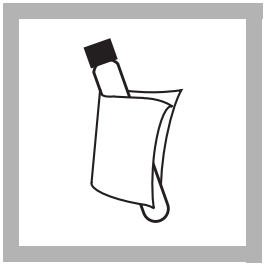


7. Carefully remove the vial from the reactor. Place it in a test tube rack and allow to cool to room temperature.

Note: *Vials will be hot.*

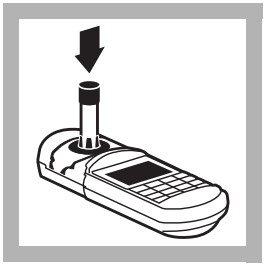


8. Remove the cap from the vial. Use a TenSette Pipet to add 2.0 mL of 1.00 N sodium hydroxide to the vial. Cap and mix.



9. Clean the outside of the vial with a towel.

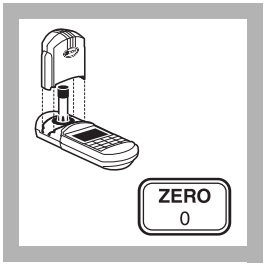
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



10. Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



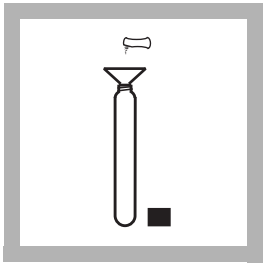
11. Tightly cover the vial with the instrument cap.

Press: **ZERO**

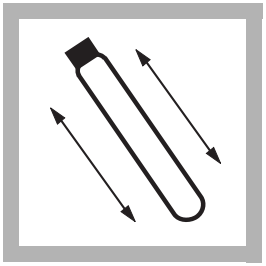
The cursor will move to the right, then the display will show:

0.00 mg/L PO4

Note: *For multiple samples, zero on the first sample. Read the remaining samples after adding the PhosVer 3 reagent. Subtract the reagent blank value from each reading.*

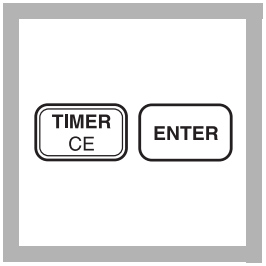


12. Remove the cap from the vial. Using a funnel, add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to the vial.



13. Cap tightly and shake for 10-15 seconds.

Note: *The powder will not completely dissolve.*



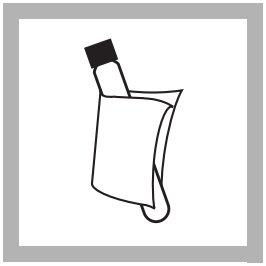
14. Press:

TIMER ENTER

A 2-minute reaction period will begin.

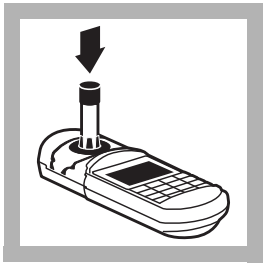
Note: *Read samples between 2 and 8 minutes after adding the PhosVer 3 reagent.*

Note: *A blue color will form if phosphate is present.*



15. After the timer beeps, clean the outside of the sample vial with a towel.

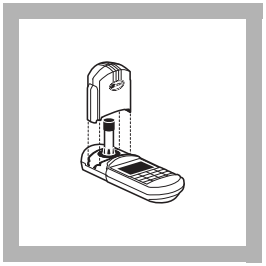
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



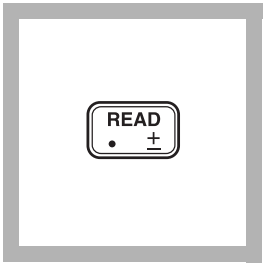
16. Place the prepared sample in the adapter

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



17. Tightly cover the vial with the instrument cap.



18. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water.
Do not use commercial detergents containing phosphate for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, the sample may be preserved up to 48 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

Accuracy Check

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse with deionized water. Do not use detergents containing phosphate to clean glassware.*

Standard Additions Method

a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as PO_4^{3-}.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.

d) Analyze each sample as described in the procedure. Use 5.0 mL of the prepared standard additions for each test; the concentration should increase as follows: 0.2 mg/L, 0.4 mg/L, and 0.6 mg/L PO_4^{3-}, respectively.

e) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Obtain a 1.0 mg/L Phosphate Standard Solution listed under *Optional Reagents*. Or, this can be prepared by pipetting 2 mL of a Voluette Ampule Standard for Phosphate, 50 mg/L as PO_4^{3-}, into an acid washed Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Substance	Interference Level and Treatment
Aluminum	200 mg/L
Arsenate	Interferes at any level.
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Sulfide	9 mg/L. Sulfide interference may be removed by oxidation with Bromine Water as follows: 1. Measure 25 mL of sample into a 50-mL beaker. 2. Swirling constantly, add Bromine Water drop-wise until a permanent yellow color develops. 3. Swirling constantly, add Phenol Solution dropwise until the yellow color just disappears. Proceed with *step 1*.

Turbidity (large amounts)	May cause inconsistent results because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences (Section 1).

The PhosVer 3 Phosphate Reagent Powder Pillows should be stored in a cool, dry environment.

Method Performance

Precision

In a single laboratory, using a standard solution of 3.00 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.06 mg/L PO_4^{3-}.

Estimated Detection Limit

The estimated detection limit for program 82 is 0.07 mg/L PO_4^{3-}. For more information on the estimated detection limit, see *Section 1*.

Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS

Total and Acid Hydrolyzable Test 'N Tube Reagent Set ..... 50 tests 27427-45
Includes: (1) 272-42, (1) 1045-42, (1) 20847-66, (1) 21060-46, (50) Total and Acid Hydrolyzable Test Vials* (1) 27430-42

Description	Quantity Required Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows	1	50/pkg	21060-46
Potassium Persulfate powder Pillows	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.0 N	2 mL	100 mL	1045-42
Total and Acid Hydrolyzable Test Vials	1	50/pkg	*
Water, deionized for reagent blanks	5 mL	100 mL	272-42

REQUIRED APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
COD/TNT Adapter	1	each	48464-00
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Funnel, micro	1	each	25843-35
Pipet, TenSette, 1 to 10 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet		50/pkg	21997-96
Test Tube Rack	1-3	each	18641-00

OPTIONAL REAGENTS

Bromine Water, 30 g/L ..... 29 mL 2211-20
Drinking Water Standard, Inorganic, F^-, $NO_3^{-N,}$ PO_4^{3-}, SO_4^{2-} 500mL 28330-49
Hydrochloric Acid Standard Solution, 6.0 N (1:1) ..... 500 mL 884-49
Phenol Solution, 30 g/L ..... 29 mL 2112-20
Phosphate Standard Solution, 1 mg/L as PO_4^{3-} ..... 500 mL 2569-49
Phosphate Standard Solution, PourRite ampule,
50 mg/L as PO_4^{3-}, 2 mL ..... 20/pkg 171-20H
Phosphate Standard Solution, Voluette ampule,
50 mg/L as PO_4^{3-}, 10 mL 16/pkg 171-10
Sodium Hydroxide Standard Solution, 5.000 N ..... 1000 mL 2450-53
Sulfuric Acid Standard Solution, 1.000 N ..... 1 L 1270-53
Wastewater Effluent Standard, Inorganic
(NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC) ..... 500 mL 28332-49
Water, deionized ..... 4 L 272-56

* These items are not sold separately.

OPTIONAL APPARATUS

Description	Units	Cat. No.
Ampule Breaker Kit, Voluette	each	21968-00
Ampule Breaker, PourRite	each	24846-00
Cylinder, graduated, mixing, 25 mL (3 required)	each	20886-40
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm		LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm		LTV082.52.30001
Flask, volumetric, Class A, 100 mL	each	14574-42
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion***™*1*, portable with electrode	each	51700-10
Pipet, TenSette, 0.1-1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet Filler, safety bulb	each	14651-00

For Technical Assistance, Price and Ordering

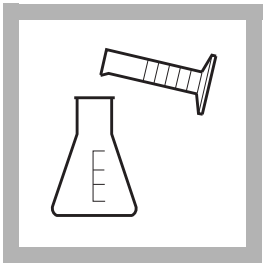
In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, TOTAL

(Also called Organic and Acid Hydrolyzable) Acid Persulfate Digestion Method*

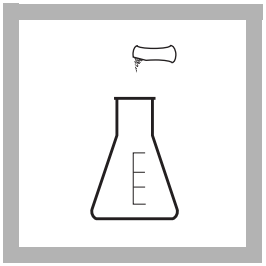
USEPA Accepted for reporting wastewater analysis**



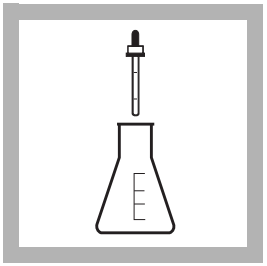
1. Measure 25 mL of sample into a 50-mL erlenmeyer flask using a graduated cylinder.

Note: *Rinse all glassware with 1:1 Hydrochloric Acid Solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

Note: *Adjust the pH of stored samples before digestion.*



2. Add the contents of one Potassium Persulfate Powder Pillow. Swirl to mix.



3. Add 2.0 mL of 5.25 N Sulfuric Acid Solution.

Note: *Use the 1-mL calibrated dropper provided.*

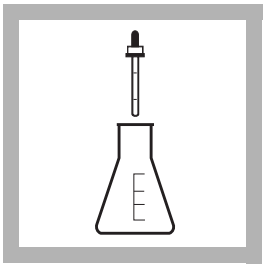
4. Place the flask on a hot plate. Boil gently for 30 minutes.

Note: *Samples should be concentrated to less than 20 mL for best recovery. After concentration, maintain the volume near 20 mL by adding small amounts of deionized water. Do not exceed 20 mL.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

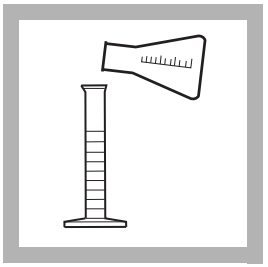
** Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P B,5 & P E.

5. Cool the sample to room temperature.



6. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution. Swirl to mix.

Note: *Use the 1-mL calibrated dropper provided.*



7. Pour the sample into a 25-mL graduated cylinder. Return the volume to 25 mL. Proceed with a reactive phosphorus test of the expected total phosphorus concentration range.

Note: *Use deionized water rinsings from the flask to adjust the volume.*

Note: *Results of the reactive phosphorus test at this point will include the organic phosphate plus the orthophosphate and the acid-hydrolyzable (condensed) phosphate. The organic phosphate concentration is determined by subtracting results of an acid hydrolyzable phosphorus test from this result. Make sure that both results are in the same units before taking the difference.*

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid-washed with 1:1 HCl and rinsed with deionized water. Do not use detergents containing phosphates for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve samples up to 28 days by adjusting the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter) and storing at 4 °C. Warm to room temperature before testing. Correct results for volume additions; see *Volume Additions* (Section 1) for more information.

Interferences

For turbid samples, use 50 mL of sample and double the reagent quantities. Use digested sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed by this procedure. For alkaline or highly buffered samples it may be necessary to add additional acid in Step 3 to drop the pH of the solution below 1.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Organically bound phosphates are thus determined indirectly by subtracting the result of an acid hydrolyzable phosphorus test from the total phosphorus result.

This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determination of the phosphorus content of the sample. If the ascorbic acid (PhosVer 3) method is used to measure the reactive phosphorus, this method is EPA approved for NPDES reporting.

The following reagents and apparatus are required in addition to those required for the reactive phosphorus test.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Potassium Persulfate Powder Pillows	1 pillow	100/pkg	2451-99
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL* MDB	2450-32
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL* MDB	2449-32

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	2	each	508-40
Flask, erlenmeyer, 50 mL	1	each	505-41
Sample Cell, 10-20-25 mL, w/caps	2	6/pkg	24019-06

OPTIONAL REAGENTS

Drinking Water Standard, Inorganic, F^-, $NO_3^{-N,}$ PO_4^{3-}, SO_4^{2-}		500mL	28330-49
Hydrochloric Acid, 6 N		500 mL	884-49
Sodium Hydroxide Solution, 5.0 N		1 L	2450-53
Sulfuric Acid		500 mL	979-49
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28332-49
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)		500 mL	28331-49
Water, deionized		4 L	272-56

* Marked Dropper Bottle - Contact Hach for larger sizes.

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Cylinder, graduated, 50 mL	each	508-41
Flask, erlenmeyer, 125 mL	each	505-43
Hot Plate, 4" diameter, 120 Vac	each	12067-01
Hot Plate, 4" diameter, 240 Vac	each	12067-02
Pads, cooling, 4 x 4"	each	18376-00
pH Indicator Paper, 1 to 11 pH	5 rolls/pkg	391-33
pH Meter, *Sens**ion***™*1*, portable with electrode	each	51700-10

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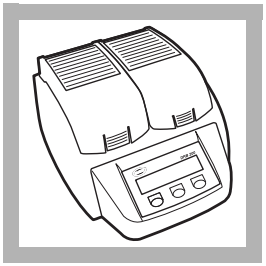
Outside the U.S.A.—Contact the Hach office or distributor serving you.

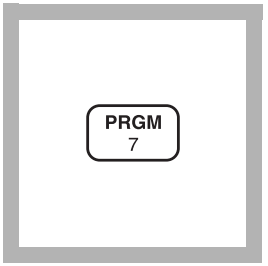
PHOSPHORUS, TOTAL (0.00 to 3.50 mg/L PO_4^{3-}) For water, wastewater and seawater

PhosVer 3 with Acid Persulfate Digestion* USEPA Accepted for reporting wastewater analysis** Test 'N Tube Procedure



1. Turn on the DRB200 Reactor. Heat the reactor to 150 °C.

Note: *See DRB200 instrument manual for selecting preprogrammed temperature applications.*



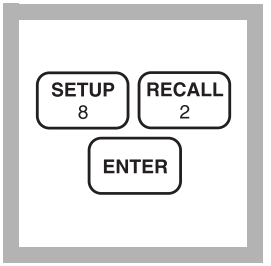
2. Enter the stored program number for total phosphorus, (PO_4^{3-}), Test 'N Tube.

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



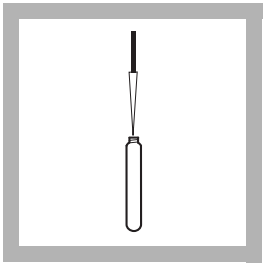
3. Press: **82 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon**.**

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*

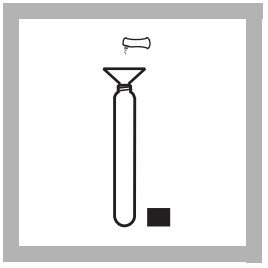
4. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

Note: *A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*

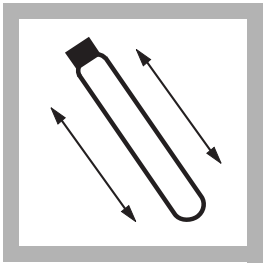


5. Use a TenSette Pipet to add 5.0 mL of sample to a Total and Acid Hydrolyzable Test Vial.

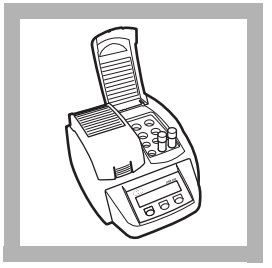
Note: *Adjust the pH of stored samples to 6-8 before analysis.*



6. Using a funnel, add the contents of one Potassium Persulfate Powder Pillow for Phosphonate to the vial.



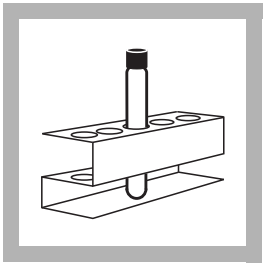
7. Cap tightly and shake to dissolve.



8. Place the vial in the DRB200 Reactor. Heat the vial for 30 minutes.

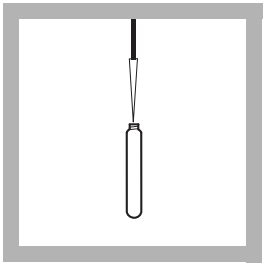
* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

** Procedure is equivalent to USEPA Method 365.2 and Standard Method 4500-P B, 5 and P.E.

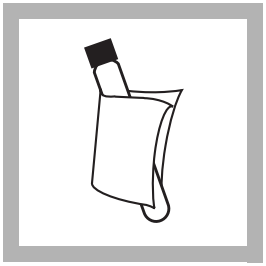


9. Carefully remove the vial from the reactor. Place it in a test tube rack and allow to cool to room temperature.

Note: *Vials will be hot.*

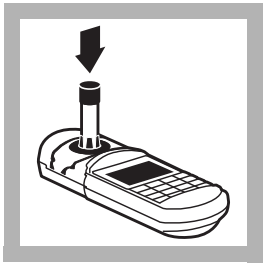


10. Use a TenSette Pipet to add 2.0 mL of 1.54 N sodium hydroxide to the vial. Cap and mix.



11. Clean the outside of the vial with a towel.

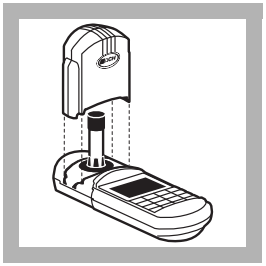
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



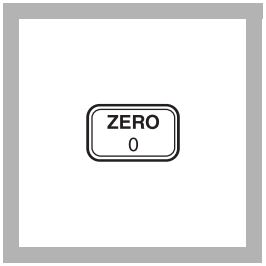
12. Place the sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



13. Tightly cover the vial with the instrument cap.

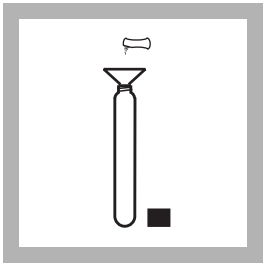


14. Press: **ZERO**

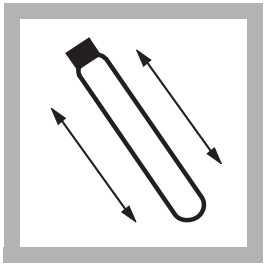
The cursor will move to the right, then the display will show:

0.00 mg/L PO4

Note: *For multiple samples, zero only on the first sample. Read the remaining samples after adding the PhosVer 3 reagent.*

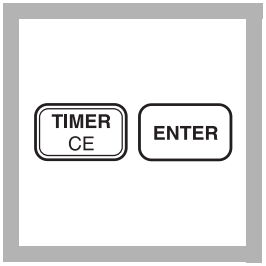


15. Remove the cap from the vial. Using a funnel, add the contents of one PhosVer 3 Phosphate Reagent Powder Pillow to the vial.



16. Cap tightly and shake for 10-15 seconds.

Note: *The powder will not completely dissolve.*



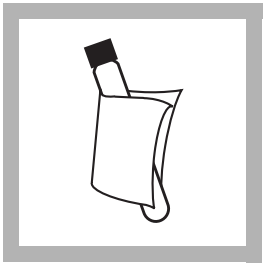
17. Press:

TIMER ENTER

A 2-minute waiting period will begin.

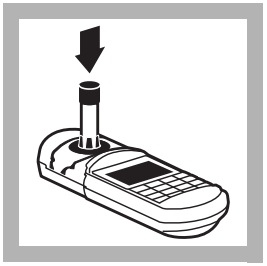
Note: *Read samples between 2 and 8 minutes after the addition of the PhosVer 3 reagent.*

Note: *A blue color will form if phosphate is present.*



18. After the timer beeps, clean the outside of the sample vial with a towel.

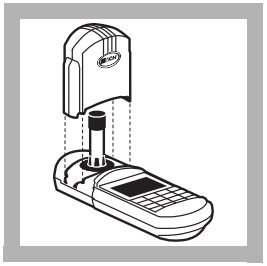
Note: *Wiping with a damp towel, followed by a dry one, will remove fingerprints or other marks.*



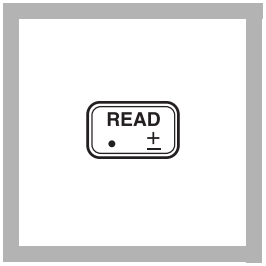
19. Place the prepared sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



20. Tightly cover the vial with the instrument cap.



21. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section 1).*

IMPORTANT NOTE:

The test range for total phosphate is limited to 0 to 3.5 mg/L PO_4^{3-}. Values above 3.5 mg/L may be used to estimate dilution ratios, but should NOT be used for reporting purposes. If a value above 3.5 mg/L PO_4^{3-} is obtained, dilute the sample and repeat the digestion and the colorimetric test.

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphates for cleaning glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve the sample for up to 28 days by adjusting the pH to 2 or less with concentrated sulfuric acid (about 2 mL per liter) and storing at 4 °C. Neutralize and warm the sample to room temperature before analysis. Correct test results for volume additions; see *Volume Additions* in *Section 1*.

Accuracy Check

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

Standard Additions Method

a) Fill three 25 mL graduated mixing cylinders with 25 mL of sample.

b) Snap the neck off a Phosphate PourRite Ampule Standard, 50 mg/L as PO_4^{3-}.

c) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 25-mL aliquots of sample prepared in *step a*. Mix well.

d) Analyze each sample as described in the procedure using 5.0 mL of the prepared standard additions for each test. The concentration should increase 0.2 mg/L, 0.4 mg/L, and 0.6 mg/L PO_4^{3-}, respectively.

e) If these increases do not occur, see *Standard Additions* (Section 1).

Standard Solution Method

To check accuracy, use a 1.0 mg/L Phosphate Standard Solution (see Optional Reagents). Or, prepare a standard by pipetting 2 mL of solution a Voluette Ampule Standard for Phosphate Standard, 50 mg/L as PO_4^{3-}, into an acid-cleaned Class A 100-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described. The mg/L PO_4^{3-} reading should be 1.0 mg/L.

OR

Prepare a 2.5 mg/L standard solution by pipetting 5 mL of a 50-mg/L Phosphate Voluette Ampule Standard into an

acid-washed 100-mL Class A volumetric flask. Dilute to the mark with deionized water.

Method Performance

Precision

In a single laboratory, using a standard solution of 3.00 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.06 mg/L PO_4^{3-}.

Estimated Detection Limit

The estimated detection limit for program 82 is 0.07 mg/L PO_4^{3-}. For more information on the estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below:

Substance	Interference Level and Treatment
Aluminum	200 mg/L
Arsenate	Interferes at any level.
Chromium	100 mg/L
Copper	10 mg/L
Iron	100 mg/L
Nickel	300 mg/L
Silica	50 mg/L
Silicate	10 mg/L
Sulfide	90 mg/L
Turbidity (large amounts)	May cause inconsistent results because the acid present in the powder pillows may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.
Zinc	80 mg/L
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see pH Interferences (Section 1).

Store PhosVer 3 Reagent Powder Pillows in a cool, dry environment.

Sample Disposal Information

Final samples will contain molybdenum. In addition, final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. Ascorbic acid then reduces the complex, giving an intense molybdenum blue color.

REQUIRED REAGENTS

Total Phosphorus Test 'N Tube Reagent Set ..... 50 tests 27426-45
Includes: (1) 272-42, (1) 20847-66, (1) 21060-46, (1) 27430-42, (50) Acid Dilution Vials*

Description	Quantity Required Per Test	Unit	Cat. No.
PhosVer 3 Phosphate Reagent Powder Pillows	1	50/pkg	21060-46
Potassium Persulfate powder Pillows	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.54 N	2 mL	100 mL	27430-42
Test 'N Tube Acid Dilution Vials	1	50/pkg	*
Water, deionized for reagent blank	5 mL	100 mL	272-42

REQUIRED APPARATUS

COD/TNT Adapter	1	each	48464-00
DRB 200 Reactor, 110 V, 15 x 16 mm tubes			LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes			LTV082.52.40001
Funnel, micro	1	each	25843-35
Test Tube Rack	1-3	each	18641-00
Pipet, TenSette, 1 to 10 mL	1	each	19700-10
Pipet Tips, for 19700-10 TenSette Pipet	varies	50/pkg	21997-96

* These items are not sold separately.

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Drinking Water Standard, Inorganic, F^-, NO_3^{-N}, PO_4^{3-}, SO_4^{2-}	500mL	28330-49
Total and Acid Hydrolyzable Test 'N Tube Reagent Set	each	27427-45
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phosphate Standard Solution, 1 mg/L as PO_4^{3-}	500 mL	2569-49
Phosphate Standard Solution, PourRite ampule, 50 mg/L as PO_4^{3-}, 2 mL	20/pkg	171-20H
Phosphate Standard Solution, Voluette ampule, 50 mg/L as PO_4^{3-}, 10 mL	16/pkg	171-10
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53
Total and Acid Hydrolyzable Test 'N Tube Reagent Set	each	27427-45
Wastewater Effluent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28332-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Ampule Breaker, PourRite ampules	each	24846-00
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm		LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm		LTV082.52.30001
Cylinder, graduated, mixing, 25 mL (3 required)	each	20886-40
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *Sens**ion**™1*, portable with electrodes	each	51700-10
Pipet Filler, safety bulb	each	14651-00
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet, TenSette, 0.1-1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28

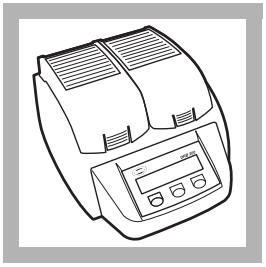
For Technical Assistance, Price and Ordering

In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

PHOSPHORUS, TOTAL, HR (0.0 to 100.0 mg/L PO_4^{3-})

Molybdovanadate Method with Acid Persulfate Digestion*
Test 'N Tube™ Procedure **For water and wastewater**



1. Turn on the DRB200 Reactor. Heat to 150 °C.

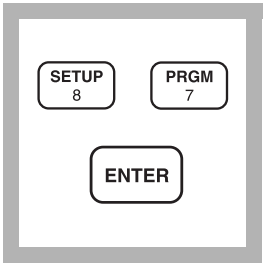
Note: *See DRB200 instrument manual for selecting preprogrammed temperature applications*

2. Enter the stored program number for phosphorus total high range, Test 'N `Tube.

Press: **PRGM**

The display will show:

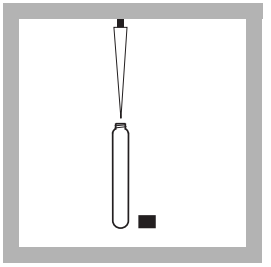
PRGM ?



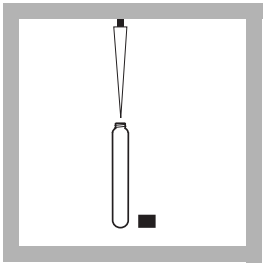
3. Press: **87 ENTER**

The display will show **mg/L, PO4** and the **ZERO** icon.

Note: *For alternate forms (P, P_2O_5), press the* **CONC** *key.*

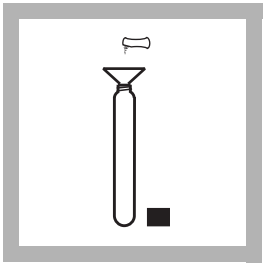


4. Use a TenSette® Pipet to add 5.0 mL of deionized water to a Total Phosphorus Test 'N Tube Vial (the blank).

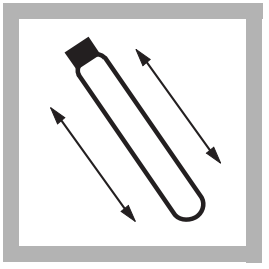


5. Use a TenSette Pipet to add 5.0 mL of sample to a Total Phosphorus Test 'N Tube Vial (the sample).

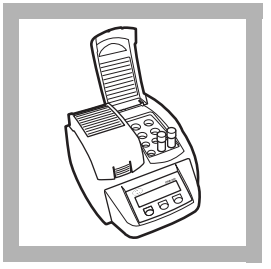
Note: *Adjust the pH of stored samples to 6–8 before analysis.*



6. Use a funnel to add the contents of one Potassium Persulfate Powder Pillow for Phosphonate to each vial.



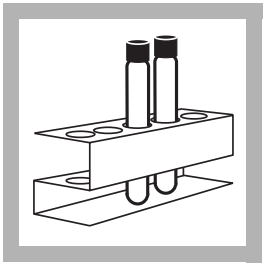
7. Cap tightly and shake to dissolve.



8. Place the vials in the DRB200 Reactor. Heat for 30 minutes.

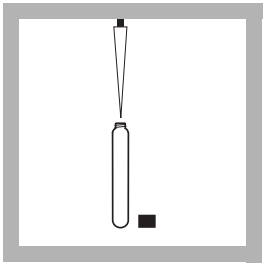
Press: **TIMER ENTER** to time the heating period.

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



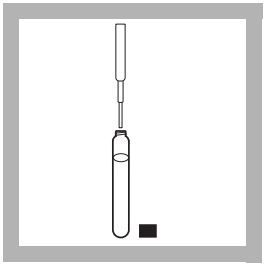
9. Carefully remove the vials from the reactor. Place them in a test tube rack and allow to cool to room temperature (18–25 °C).

Note: *Vials will be hot.*



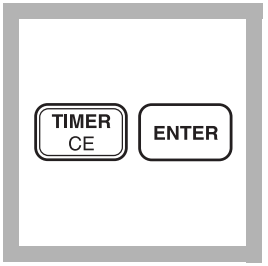
10. Use a TenSette Pipet to add 2.0 mL of 1.54 N sodium hydroxide to each vial.

Cap and invert to mix.



11. Use a polyethylene dropper to add 0.5 mL of Molybdovanadate Reagent to each vial.

Cap and invert to mix.



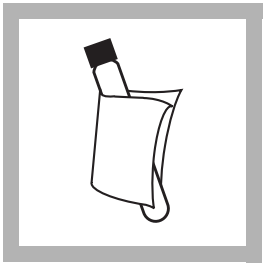
12. Press:

TIMER ENTER

A 7-minute reaction period will begin.

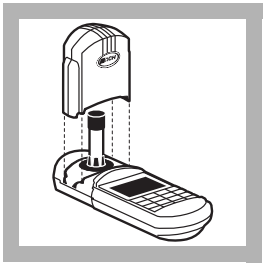
Note: *Read the samples between 7 and 9 minutes.*

13. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.



14. Clean the outside of the vials with a towel.

Note: *Wipe with a damp towel, followed by a dry one, to remove fingerprints or other marks.*

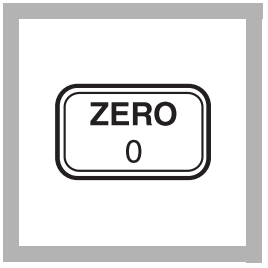


15. When the timer sounds, place the blank vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Tightly cover the vial with the instrument cap.

Note: *Do not move the vial from side to side as this can cause errors.*

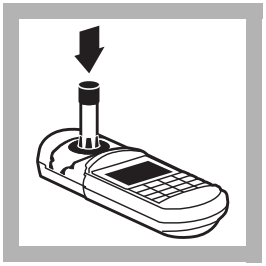


16. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L PO4

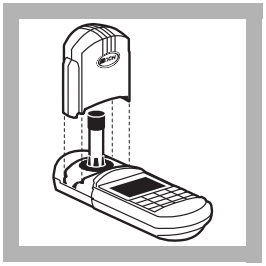
Note: *Reagent blanks for each lot of reagents may be used more than once, but should not be used for longer than one day.*



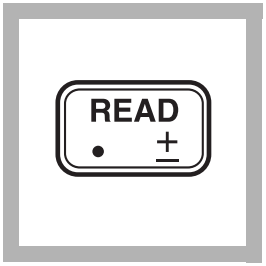
17. Place the prepared sample vial in the adapter.

Push straight down on the top of the vial until it seats solidly into the adapter.

Note: *Do not move the vial from side to side as this can cause errors.*



18. Tightly cover the vial with the instrument cap.



19. Press: **READ**

The cursor will move to the right, then the result in mg/L phosphate (PO_4^{3-}) will be displayed.

Note: *For best results, use Standard Adjust with each new lot of reagent. (See* Accuracy Check.*)*

Sampling and Storage

Collect samples in plastic or glass bottles that have been acid cleaned with 1:1 Hydrochloric Acid Solution and rinsed with deionized water. Do not use commercial detergents containing phosphates for cleaning the glassware used in this test.

Analyze samples immediately after collection for best results. If prompt analysis is impossible, preserve the sample for up to 28 days by adjusting the pH to 2 or less with concentrated H_2SO_4 (about 2 mL per liter) and storing at 4 °C. Warm the sample to room temperature and neutralize with 5.0 N NaOH before analysis.

Correct test results for volume additions; see *Volume Additions* in *Section 1*of the *DR/890 Procedures Manual*.

Accuracy Check

Note: *Clean glassware with 1:1 hydrochloric acid solution. Rinse again with deionized water. Do not use detergents containing phosphates to clean glassware.*

Standard Additions Method

a. Fill each of three 10-mL graduated mixing cylinders with 10 mL of sample.

b. Snap the neck off a 10-mL Voluette® Ampule of Phosphate Standard Solution, 500 mg/L as PO_4^{3-} (Cat. No. 14242-10).

c. Use a TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL, respectively, to the three 10-mL aliquots of the water sample prepared in *step a*. Mix well.

d. Analyze samples from *step c* as described in the procedure. Use 5.0 mL of the prepared sample for each test. The concentration should increase: 5 mg/L, 10 mg/L, and 15 mg/L PO_4^{3-}, respectively.

e. If these increases do not occur, see *Standard Additions (Section 1 of the DR/890 Procedures Manual*) for more information.

Standard Solution Method

To check accuracy, prepare an 80 mg/L standard by pipetting 8.0 mL of solution from a 10-mL Voluette® Ampule of Phosphate Standard Solution, 500 mg/L as PO_4^{3-} into an acid-cleaned, Class A, 50-mL volumetric flask. Dilute to the mark with deionized water. Substitute this standard for the sample and perform the procedure as described.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 80 mg/L PO_4^{3-} standard solution, press the **SETUP** key and scroll, using the arrow keys, to the **STO** option. Press **ENTER** to activate the standard adjust option. Then enter 80.0 to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Standard Curve Adjustment, Section 1* of the *Procedures Manual* for more information.

Interferences

Large amounts of sample turbidity may cause inconsistent results in the test because the acid present in the reagents may dissolve some of the suspended particles and because of variable desorption of orthophosphate from the particles.

The following may interfere when present in concentrations exceeding those listed below:

Interfering Substance	Interference Level and Treatment
Arsenate	Causes positive interference if the sample is heated.[1]
Iron, ferrous	Blue color caused by ferrous iron does not interfere if iron concentration is less than 100 mg/L.
Molybdate	Causes negative interference above 1000 mg/L.
Silica	Causes positive interference if the sample is heated.*
Extreme pH or highly buffered samples	May exceed buffering capacity of the reagents. See *pH Interferences* in *Section 1* of the *DR/890 Procedures Manual*. Samples may require pretreatment. Sample pH should be about 7.
Fluoride, thorium, bismuth, thiosulfate or thiocyanate	Cause a negative interference.
Temperature, Cold (less than 18 °C)	Causes a negative interference.
Temperature, Hot (greater than 25 °C)	Causes a positive interference. Post-digestion samples should be brought to room temperature (18–25 °C) before the addition of the Molybdovanadate Reagent or sodium hydroxide.
The following do not interfere in concentrations up to 1000 mg/L: Pyrophosphate, tetraborate, selenate, benzoate, citrate, oxalate, lactate, tartrate, formate, salicylate, Al^{3+}, Fe^{3+}, Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+}, Li^{+}, Na^{+}, K^{+}, NH_4^{+}, Cd^{2+}, Mn^{2+}, NO_3^{-}, NO_2^{-}, SO_4^{2-}, SO_3^{2-}, Pb^{2+}, Hg^{+}, Hg^{2+}, Sn^{2+}, Cu^{2+}, Ni^{2+}, Ag^{+}, U^{4+}, Zr^{4+}, AsO_3^{-}, Br^{-}, CO_3^{2-}, ClO_4^{-}, CN^{-}, IO_3^{-}, SiO_4^{4-}.	

[1] Gentle warming of the sample to reach room temperature will not cause this substance to interfere.

Method Performance

Precision

In a single laboratory, using a standard solution of 80.0 mg/L PO_4^{3-} and two lots of reagent with the instrument, a single operator obtained a standard deviation of ±3.0 mg/L PO_4^{3-}.

Estimated Detection Limit

The estimated detection limit for program 87 is 7.0 mg/L PO_4^{3-}. For more information on the estimated detection limit, see *Section 1* of the *DR/890 Procedures Manual*.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet for information specific to the reagents used.

Sample Disposal Information

The final samples will contain molybdenum. In addition, the final samples will have a pH less than 2 and are considered corrosive (D002) by the Federal RCRA. Consult the Material Safety Data Sheet for information specific to the reagent used.

Summary of Method

Phosphates present in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate.

Orthophosphate reacts with molybdate in an acid medium to produce a phosphomolybdate complex. In the presence of vanadium, yellow vanadomolybdophosphoric acid forms. The intensity of the yellow color is proportional to the phosphate concentration.

Installing this Program on the DR/800

This procedure will add the current method as a new Hach program to your DR/800.

1. Turn the DR/800 on by pressing the **ON** key.
2. Press the **SETUP** key.
3. Press the down arrow key two times so that the prompt line shows **USER**.
4. Press the **ENTER** key.
5. Enter **8138**, followed by **ENTER**.
6. Enter each of the numbers in the right column, each followed by **ENTER**. The line numbers in the left column relate to the line number on the display. At any time you may use the arrow keys to scroll back to review or change any number you have already entered.

Line Number	Entry	Line Number	Entry
1	87	18	0
2	4	19	0
3	73	20	80
4	0	21	79
5	0	22	52
6	0	23	0
7	0	24	0
8	0	25	80
9	0	26	0
10	0	27	0
11	0	28	0
12	66	29	0
13	175	30	80
14	48	31	50
15	32	32	79
16	0	33	53
17	0	34	0
35	62	46	0

Line Number	Entry	Line Number	Entry
36	166	47	15
37	246	48	7
38	148	49	8
39	63	50	1
40	63	51	164
41	78	52	0
42	252	53	0
43	4	54	40
44	76	55	0
45	128	56	255

REQUIRED REAGENTS

Total High Range Phosphorus Test 'N Tube™ Reagent Set ..... 50 vials 27672-45
Includes: (50) Total Phosphorus Test 'N Tube™ Vials*, (2) 272-42, (1) 20847-66
(1) 20760-26, (1) 27430-42

Description	Quantity Required Per Test	Unit	Cat. No.
Molybdovanadate Reagent	0.5 mL	25 mL	20760-26
Potassium Persulfate Powder Pillows	1	50/pkg	20847-66
Sodium Hydroxide Solution, 1.54 N	2 mL	100 mL	27430-42
Total Phosphorus Test 'N Tube™ Vials	1	50/pkg	*
Water, deionized		100 mL	272-42

REQUIRED APPARATUS

DRB 200 Reactor, 110 V, 15 x 16 mm tubes LTV082.53.40001
DRB 200 Reactor, 220 V, 15 x 16 mm tubes LTV082.52.40001
COD/TNT Adapter, DR/800 series ..... 1 ..... each 48464-00
Dropper, LDPE, 0.5 to 1.0 mL ..... 1 ..... 20/pkg 21247-20
Pipet, TenSette®, 1 to 10 mL ..... 1 ..... each 19700-10
Pipet Tips, for 19700-10 TenSette® Pipet ..... varies .. 50/pkg 21997-96
Test Tube Rack ..... 1–3 ..... each 18641-00

* These items are not sold separately.

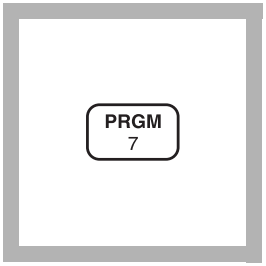
OPTIONAL REAGENTS

Description	Unit	Cat. No.
Hydrochloric Acid Standard Solution, 6.0 N (1:1)	500 mL	884-49
Phosphate Standard Solution, PourRite™ ampule, 500 mg/L as PO_4^{3-}, 2-mL	20/pkg	14242-20
Phosphate Standard Solution, Voluette™ ampule, 500 mg/L as PO_4^{3-}, 10-mL	16/pkg	14242-10
Sodium Hydroxide Standard Solution, 5.0 N	1 L	2450-53
Sulfuric Acid, ACS, concentrated	500 mL	979-491
Wastewater Influent Standard, Inorganic (NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC)	500 mL	28331-49

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Aspirator, vacuum	each	2131-00
Cylinder, graduated, mixing, 10 mL (3 required)	each	20886-38
DRB 200 Reactor, 110 V, 21 x 16 mm and 4 x 20 mm		LTV082.53.42001
DRB 200 Reactor, 220 V, 21 x 16 mm and 4 x 20 mm		LTV082.52.42001
DRB 200 Reactor, 110 V, 9 x 16 mm and 2 x 20 mm		LTV082.53.30001
DRB 200 Reactor, 220 V, 9 x 16 mm and 2 x 20 mm		LTV082.52.30001
Filter Holder, 47 mm, 300 mL, graduated	each	13529-00
Filter, membrane, 47 mm, 0.45 microns	200/pkg	13530-01
Flask, filtering, 500-mL	each	546-49
Flask, volumetric, Class A, 50-mL	each	14574-41
pH Indicator Paper, 1 to 11 pH units	5 rolls/pkg	391-33
pH Meter, *sens**ion**™1*, portable with electrode	each	51700-10
Pipet Filler, Safety Bulb	each	14651-00
Pipet, TenSette®, 0.- to 1.0-mL	each	19700-01
Pipet Tips, for 19700-01	50/pkg	21856-96
Pipet Tips, for 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 8.00-mL	each	14515-08
Stopper, No. 7 one hole	6/pkg	2119-07
Tubing, rubber	12 feet	560-19

Heteropoly Blue Method*

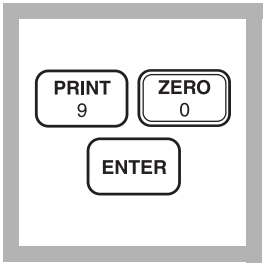


1. Enter the stored program number for low range silica (SiO_2).

Press: **PRGM**

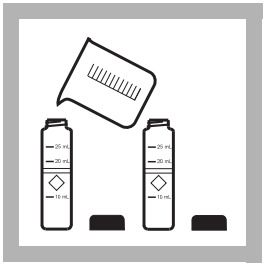
The display will show:

PRGM ?

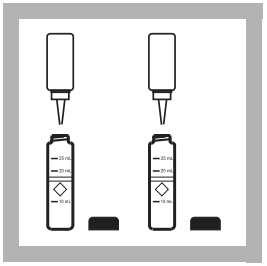


2. Press: **90 ENTER**

The display will show **mg/L, SiO2** and the **ZERO** icon**.**

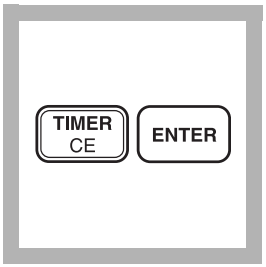


3. Fill two sample cells to the 10-mL line with sample.



4. Add 15 drops of Molybdate 3 Reagent to each sample cell. Swirl to mix.

Note: *For greatest accuracy, hold dropping bottle vertical.*

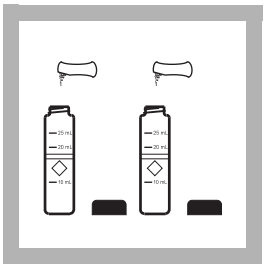


5. Press:

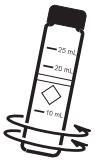
TIMER ENTER

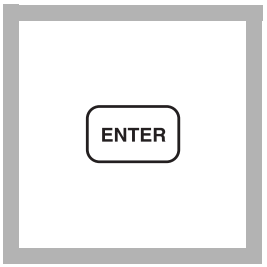
A 4-minute reaction period will begin.

Note: *Reaction time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait 8 minutes. If the sample temperature is 30 °C (86 °F), wait 2 minutes.*



6. After the timer beeps, add the contents of one Citric Acid Reagent Powder Pillow to each sample cell. Swirl to mix.





7. The display will show:

1:00 TIMER 2

Press: **ENTER**

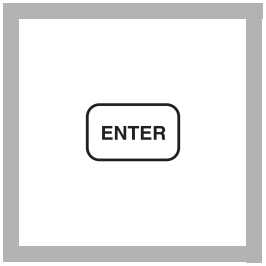
A 1-minute reaction period will begin. Phosphate interference is eliminated during this period.

Note: *The time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait two minutes. If the sample is 30 °C (86 °F), wait 30 seconds.*

8. After the timer beeps, add the contents of one Amino Acid F Reagent Powder Pillow to one of the sample cells (the prepared sample). Invert to mix.

Note: *The sample cell without the Amino Acid F Reagent is the blank.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*



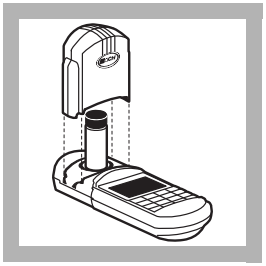
9. The display will show:

2:00 TIMER 3

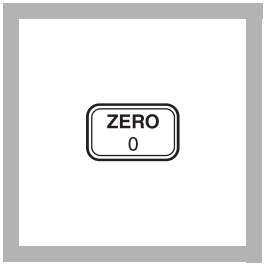
Press: **ENTER**

A 2-minute reaction period will begin.

Note: *A blue color will develop if silica is present.*



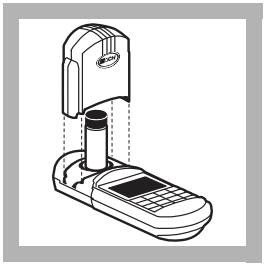
10. After the timer beeps, place the blank (solution without Amino Acid F Reagent) into the cell holder. Tightly cover the sample cell with the instrument cap.



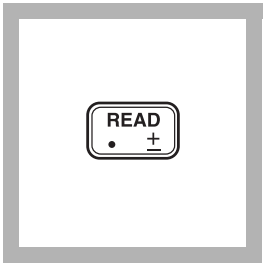
11. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L SiO2



12. Place the sample into the cell holder. Tightly cover the sample cell with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L SiO2 will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic bottles. Analyze samples as soon as possible after collection. If prompt analysis is not possible, store samples for up to 28 days by cooling to 4 °C (39 °F) or below. Warm samples to room temperature before analysis.

Accuracy Check

Standard Additions Method

a) Open a Silica Standard Solution Bottle, 25 mg/L SiO_2.

b) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard to three 10-mL samples. Mix thoroughly.

c) Analyze each sample as described above. The silica concentration should increase 0.25 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Adjust

To adjust the calibration curve using the reading obtained with the
1.00-mg/L Standard Solution (see *Optional Reagents*), press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **1.00** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1*, *Standard Curve Adjustment* for more information.

Method Performance

Precision

In a single laboratory, using standard solutions of 1.00 mg/L silica and two representative lots of reagent and a instrument, a single operator obtained a standard deviation of ±0.025 mg/L silica.

Estimated Detection Limit (EDL)

The estimated detection limit for program 90 is 0.020 mg/L SiO2. For more information on the estimated detection limit, see *Section 1*. If testing for very low levels of silica, use the ultra-low range silica method on the Hach DR/2010 or DR/4000 Spectrophotometers.

Interferences

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Phosphate	Phosphate does not interfere at levels less than 50 mg/L PO_4. At 60 mg/L PO_4, an interference of -2% occurs. At 75 mg/L PO_4 the interference is -11%.
Iron	Large amounts of iron interfere.
Slow reacting forms of silica	Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these "molybdate-unreactive" forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the Examination of Water and Wastewater* under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help in lieu of the bicarbonate pretreatment.
Sulfides	Interfere at all levels
Turbidity	Eliminated by zeroing the instrument with the original sample.

Reagent Preparation

To prepare Amino Acid F Reagent Solution, dissolve 11.4 grams of Amino Acid F Reagent Powder in 100 mL of 1.0 N Sodium Hydroxide Solution. The solution is stable for at least one month if stored in a plastic bottle.

Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Acid reduces the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration.

REQUIRED REAGENTS

	Cat. No.
Low Range Silica Reagent Set, 10 mL sample (100 tests)	24593-00
Includes: (1) 22540-69, (1) 21062-69 (2) 1995-26	

Description	Quantity Required Per Test	Units	Cat. No.
Amino Acid F Reagent Powder Pillows	1 pillow	100/pkg	22540-69
Citric Acid Powder Pillows	2 pillows	100/pkg	21062-69
Molybdate 3 Reagent	28 drops	50 mL SCDB	1995-26

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Silica Standard Solution, 1.00 mg/L SiO_2	500 mL	1106-49
Silica Standard Solution, 25 mg/L SiO_2	236 mL	21225-31
Sodium Bicarbonate, ACS	454 g	776-01
Sodium Hydroxide Standard Solution, 1.000 N	900 mL	1045-53
Sulfuric Acid Standard Solution, 1.0 N	1000 mL	1270-53

OPTIONAL APPARATUS

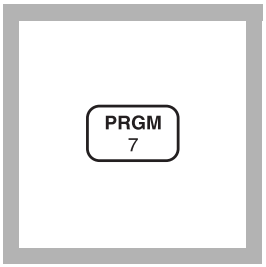
Bottle, 118 mL, polyethylene, oblong	6/pkg	23184-06
Dropper, 0.5- & 1.0-mL marks	6/pkg	23185-06
Pipet, serological, 2 mL, poly	each	2106-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 Pipet	1000/pkg	21856-28
Standard Methods for the Examination of Water and Wastewater	each	22708-00
Thermometer, - 20 to 110 °C, Non-Mercury	each	26357-02

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

Silicomolybdate Method



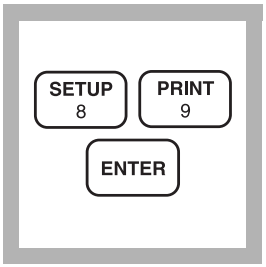
1. Enter the stored program number for high range silica (SiO_2).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **89 ENTER**

The display will show **mg/L, SiO2** and the **ZERO** icon**.**

Note: *For alternate form (Si), press the* **CONC** *key.*

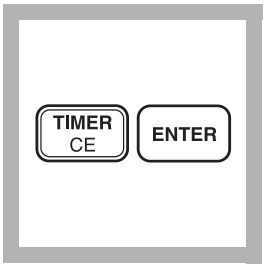
3. Fill two sample cells with 10 mL of sample. Set one aside as the blank.

Note: *Sample temperature should be 15 to 25 °C (59 to 77 °F).*

4. To the other cell, add the contents of one Molybdate Reagent Powder Pillow for High Range Silica (the prepared sample). Cap and invert to mix.

5. Add the contents of one Acid Reagent Powder Pillow for High Range Silica. Cap and invert to mix.

Note: *Silica or phosphate will cause a yellow color to develop.*



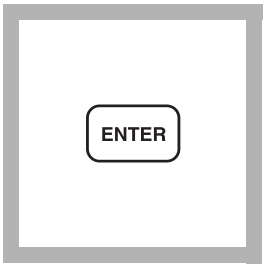
6. Press:

TIMER ENTER

A 10-minute reaction period will begin.

7. When the timer beeps, add the contents of one Citric Acid Powder Pillow to the prepared sample. Cap and invert to mix.

Note: *The yellow color due to phosphate will disappear.*

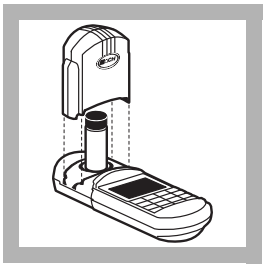


8. The display will show: **2:00 Timer 2**

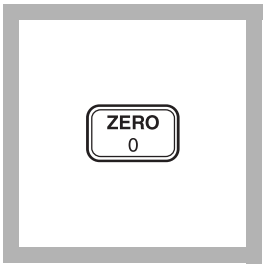
Press: **ENTER**

A two-minute reaction period will begin.

Note: *Perform Steps 9-12 within three minutes after the timer beeps.*



9. When the timer beeps, place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

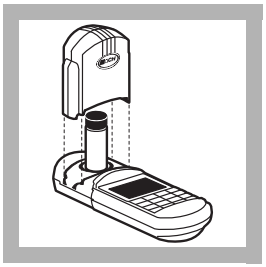


10. Press: **ZERO**

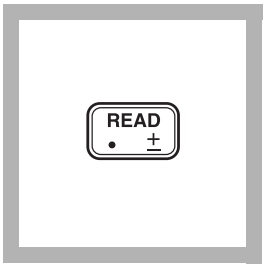
The cursor will move to the right, then the display will show:

0.0 mg/L SiO2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L silica (SiO_2) will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. Store samples up to 28 days at 4 ˚C (39 ˚F) or below. Warm samples to room temperature before analyzing.

Accuracy Check

Standard Additions Method

a) Open a High Range Silica Standard Solution, 1000 mg/L SiO_2.

b) Use the TenSette Pipet to add 0.1 mL, 0.3 mL, and 0.5 mL of the standard to three 10-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The silica concentration should increase 10.0 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To check the accuracy of the method, use the Silica Standard Solutions, 25 and 50 mg/L as SiO_2, listed under Optional Reagents. Analyze according to the above procedure using deionized water as the blank.

Standard Adjust

To adjust the calibration curve using the reading obtained with the
50.0 mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **50.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1*, *Standard Curve Adjustmen*t for more information.

Method Performance

Precision

In a single laboratory, using a standard solution of 50.0 mg/L SiO_2 and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±1.0 mg/L silica.

Estimated Detection Limit

The estimated detection limit for program 89 is 1.00 mg/L SiO_2. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Iron	High levels of Fe^{2+} and Fe^{3+} interfere.
Phosphate	Does not interfere below 50 mg/L PO_4^{3-}. At 60 mg/L PO_4^{3-}, a negative 2% interference occurs. At 75 mg/L PO_4^{3-} the interference is negative 11%.
Sulfides (S^{2-})	High levels interfere.
Turbidity	Eliminated by zeroing the instrument with the original sample.

Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these “molybdate-unreactive” forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the*

Examination of Water and Wastewater under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help in lieu of the bicarbonate treatment.

Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Silica is then determined by measuring the remaining yellow color.

REQUIRED REAGENTS

	Cat. No.
High Range Silica Reagent Set, 10-mL sample (100 tests)	24296-00
Includes: (1) 21074-69, (1) 21062-69, (1) 21073-69	

Description	Quantity Required Per Test	Units	Cat. No.
Acid Reagent Powder Pillows for High Range Silica	1	100/pkg	21074-69
Citric Acid Powder Pillows	1	100/pkg	21062-69
Molybdate Reagent Powder Pillows for HR Silica	1	100/pkg	21073-69

REQUIRED APPARATUS

Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

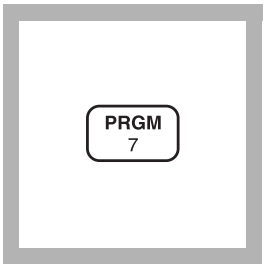
Silica Standard Solution, 10 mg/L	500 mL	1403-49
Silica Standard Solution, 25 mg/L	236 mL	21225-31
Silica Standard Solution, 50 mg/L	200 mL	1117-29
Silica Standard Solution, 1000 mg/L	500 mL	194-49
Sodium Bicarbonate, ACS	454 g	776-01
Sulfuric Acid Standard Solution, 1.000 N	100 mL MDB	1270-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 Pipet	1000/pkg	21856-28
Standard Methods for the Examination of Water and Wastewater	each	22708-00
Thermometer, -20 to 110 ˚C, Non-Mercury	each	26357-02

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

Silicomolybdate Method



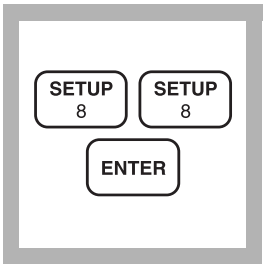
1. Enter the stored program number for ultra high range silica (SiO_2).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



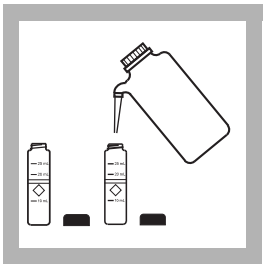
2. Press: **88 ENTER**

The display will show **mg/L, SiO2** and the **ZERO** icon**.**

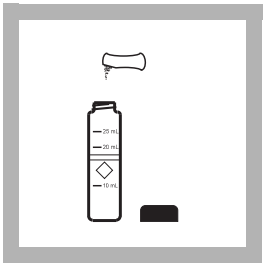
Note: *For alternate form (Si), press the* **CONC** *key.*

3. Fill 2 sample cells with 10 mL of sample.

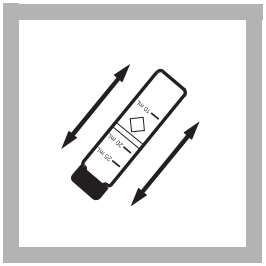
Note: *Sample temperature should be 15 to 25 °C (59 to 77 °F).*



4. Fill both sample cells to the 25-mL line with deionized water. Set one sample cell aside as the blank.

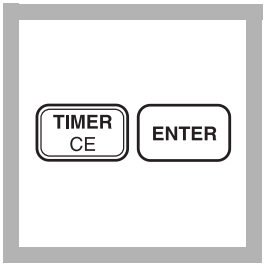


5. To the other cell, add the contents of one Molybdate Reagent Powder Pillow for High Range Silica (the prepared sample). Cap and invert to mix.



6. Add the contents of one Acid Reagent Powder Pillow for High Range Silica to the prepared sample. Cap and invert to mix.

Note: *Silica or phosphate will cause a yellow color to develop.*



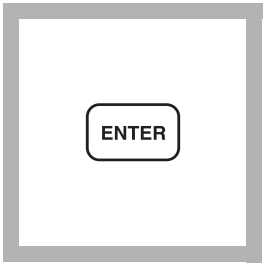
7. Press:

TIMER ENTER

A 10-minute reaction period will begin.

8. When the timer beeps, add the contents of one Citric Acid Powder Pillow to the prepared sample. Cap and invert to mix.

Note: *The yellow color due to phosphate will disappear.*

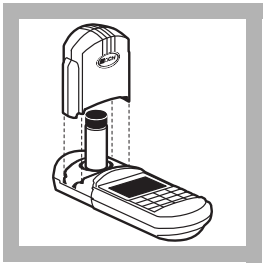


9. The display will show: **2:00 Timer 2**

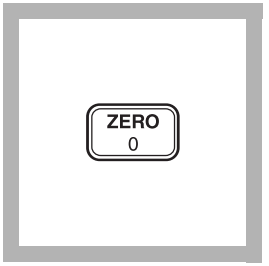
Press: **ENTER**

A two-minute reaction period will begin.

Note: *Perform Steps 10-13 within three minutes after the timer beeps.*



10. When the timer beeps, place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

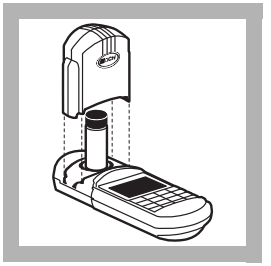


11. Press: **ZERO**

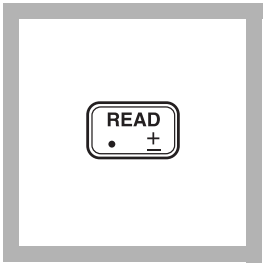
The cursor will move to the right, then the display will show:

0 mg/L SiO2

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



12. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



13. Press: **READ**

The cursor will move to the right, then the result in mg/L silica (SiO_2) will be displayed.

Note: *Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. Store samples up to 28 days at 4 °C (39 °F) or below. Warm samples to room temperature before analyzing.

Accuracy Check

Standard Additions Method

a) Open a High Range Silica Standard Solution, 1000 mg/L SiO_2.

b) Use the TenSette Pipet to add 0.1 mL, 0.3 mL, and 0.5 mL of the standard to three 10-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The silica concentration should increase 4 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

To prepare a 160-mg/L silica standard, pipet 40.0 mL of a 1000-mg/L Silica Standard Solution into a 250-mL volumetric flask. Dilute to the line with deionized water. Analyze according to the above procedure using deionized water as the blank.

Standard Adjust

To adjust the calibration curve using the reading obtained with the
160-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **160.** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1*, *Standard Curve Adjustmen*t for more information.

Method Performance

Precision

In a single laboratory, using a standard solution of 100.0 mg/L SiO_2 and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±2.0 mg/L silica.

Estimated Detection Limit

The estimated detection limit for program 88 is 3.0 mg/L SiO_2. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample.
Iron	High levels of Fe^{2+} and Fe^{3+} interfere.
Phosphate	Does not interfere below 50 mg/L PO_4^{3-}. At 60 mg/L PO_4^{3-}, a negative 2% interference occurs. At 75 mg/L PO_4^{3-} the interference is negative 11%.
Sulfides (S^{2-})	High levels interfere.
Turbidity	Eliminated by zeroing the instrument with the original sample.

Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these "molybdate-unreactive" forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in *Standard Methods for the Examination of Water and Wastewater* under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help in lieu of the bicarbonate treatment.

Summary of Method

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Silica is then determined by measuring the remaining yellow color.

REQUIRED REAGENTS

	Cat. No.
High Range Silica Reagent Set, 25-mL sample (100 tests)	22443-00
Includes: (1) 1042-99, (1) 14548-99, (1) 1041-99	

Description	Quantity Required Per Test	Units	Cat. No.
Acid Reagent Powder Pillows for High Range Silica	1	100/pkg	1042-99
Citric Acid Powder Pillows	1	100/pkg	14548-99
Molybdate Reagent Powder Pillows for HR Silica	1	100/pkg	1041-99
Water, deionized	30 mL	4 L	272-56

REQUIRED APPARATUS

Sample 10-20-15 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Silica Standard Solution, 10 mg/L	500 mL	1403-49
Silica Standard Solution, 25 mg/L	236 mL	21225-31
Silica Standard Solution, 50 mg/L	200 mL	1117-29
Silica Standard Solution, 1000 mg/L	500 mL	194-49
Sodium Bicarbonate, ACS	454 g	776-01
Sulfuric Acid Standard Solution, 1.000 N	100 mL MDB	1270-32

OPTIONAL APPARATUS

Flask, volumetric, 250 mL, Class A	each	14574-46
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 100 mL	each	14515-42
Pipet Filler, safety bulb	each	14651-00
Standard Methods for the Examination of Water and Wastewater	each	22708-00
Thermometer, -20 to 110 °C, Non-Mercury	each	26357-02

For Technical Assistance, Price and Ordering

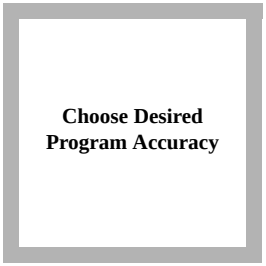
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

SULFATE (0 to 70 mg/L)

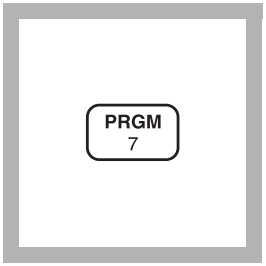
SulfaVer 4 Method* (Powder Pillows or AccuVac Ampuls); USEPA accepted for reporting wastewater analysis**

Using Powder Pillows



1. A User-Entered Calibration is necessary to obtain the most accurate results. See the *User Calibration* section at the back of this procedure. Program 91 can be used for process control or applications where a high degree of accuracy is not needed.

Note: *The nature of turbidimetric tests and reagent lot variation requires user calibration for best results.*

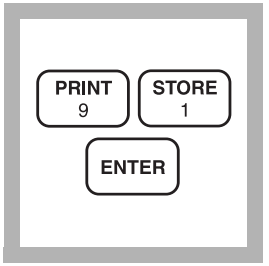


2. Enter the stored program number for sulfate (SO_4^{-}).

Press: **PRGM**

The display will show:

PRGM ?



3. Press: **91 ENTER** or the program number selected for a user-entered calibration.

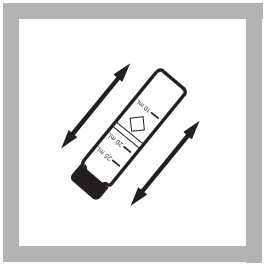
The display will show **mg/L, SO4** and the **ZERO** icon.

4. Fill a clean sample cell with 10 mL of sample.

Note: *Filter highly turbid or colored samples. Use filtered sample in this step and as the blank.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

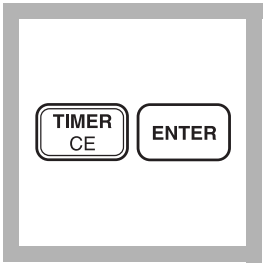
** Procedure is equivalent to USEPA method 375.4 for wastewater.



5. Add the contents of one SulfaVer 4 Sulfate Reagent Powder Pillow to the sample cell (the prepared sample). Cap the cell and invert several times to mix.

Note: *A white turbidity will develop if sulfate is present in the sample.*

Note: *Accuracy is not affected by undissolved powder.*

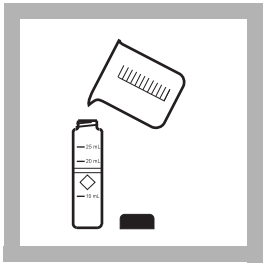


6. Press:

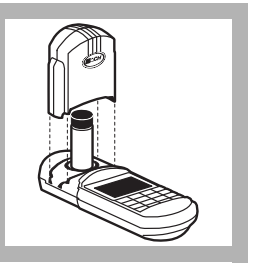
TIMER ENTER

A 5-minute reaction period will begin.

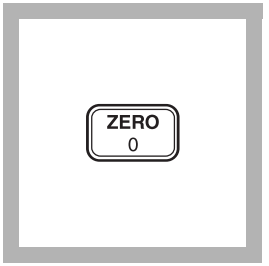
Allow the cell to stand undisturbed.



7. After the timer beeps, fill a second sample cell with 10 mL of sample (the blank).



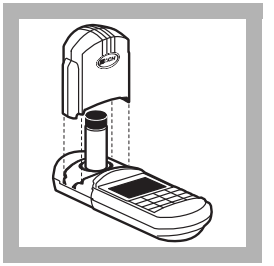
8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



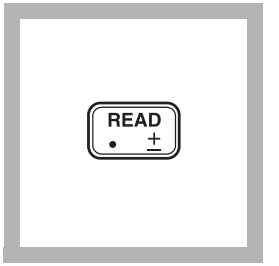
9. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L SO4



10. Within five minutes after the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



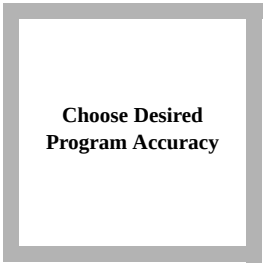
11. Press: **READ**

The cursor will move to the right, then the result in mg/L sulfate will be displayed.

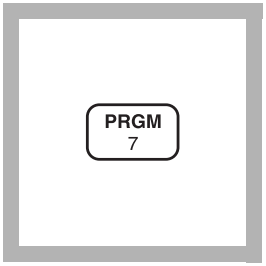
Note: *If Program 91 is used, use of the Standard Adjust is highly recommended. See Accuracy Check.*

Note: *Clean the sample cells with soap and a brush.*

Using AccuVac Ampuls



1. A User-Entered Calibration is necessary to obtain the most accurate results. See User Calibration Section at the back of this procedure. Program 92 can be used for process control or applications where a high degree of accuracy is not needed.

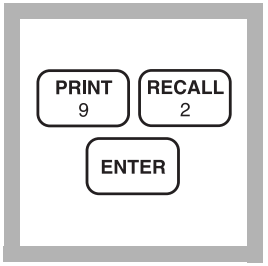


2. Enter the stored program number for sulfate (SO_4^{-})-AccuVac Ampuls.

Press: **PRGM**

The display will show:

PRGM ?

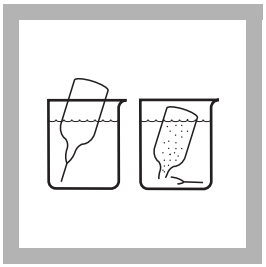


3. Press: **92 ENTER**

The display will show **mg/L, SO4** and the **ZERO** icon**.**

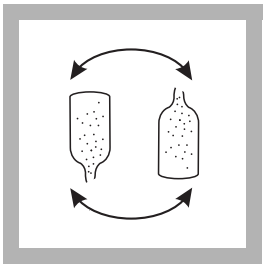
4. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

Note: *Filter highly turbid or colored samples. Use filtered sample in this step and as the blank.*



5. Fill a SulfaVer 4 Sulfate AccuVac Ampul with sample.

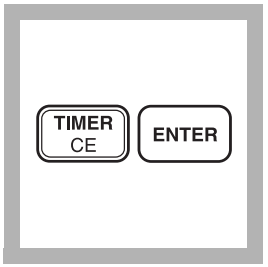
Note: *Keep tip immersed until the ampul fills completely.*



6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A white turbidity will develop if sulfate is present.*

Note: *Accuracy is not affected by undissolved powder.*

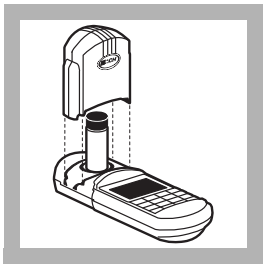


7. Press:

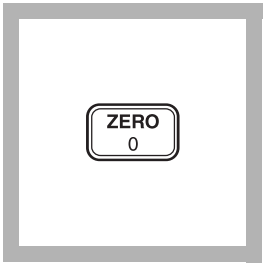
TIMER ENTER

A 5-minute reaction period will begin.

Note: *Allow the ampul to stand undisturbed.*



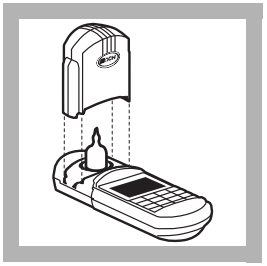
8. After the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



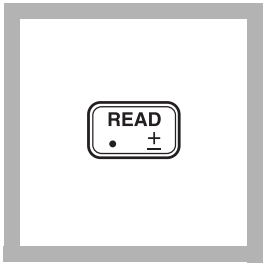
9. Press: **ZERO**

The cursor will move to the right, then the display will show:

0 mg/L SO4



10. Within five minutes after the timer beeps, place the AccuVac ampul into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L sulfate will be displayed.

Note: *If Program 92 is used, use of the Standard Adjust is highly recommended. See Accuracy Check.*

User- Entered Calibration

There are various programs to determine sulfate, each with a different level of accuracy. Best results are obtained by performing a user-entered calibration with each new lot of reagent. Programs 91 and 92 can be run when a high degree of accuracy is not needed. Use of the Standard Adjust feature will improve performance when using programs 91 and 92. It should NOT be used with a user calibration, as it will hinder performance.

Using Class A glassware, prepare standards of 10, 20, 30, 40, 50, 60, and 70 mg/L sulfate by pipetting 1, 2, 3, 4, 5, 6, and 7 mL of a 1000-mg/L sulfate standard into 100-mL volumetric flasks. Dilute to the mark with deionized water and mix well.

Zero the instrument with water. The user-entered settings for sulfate are:

Program number: #101 to 105
Wavelength: 520 nm
Resolution: 0 mg/L

See *Creating User-Entered Program* in the instrument manual for specific instructions on entering a user-entered program.

Sampling and Storage

Collect samples in clean plastic or glass bottles. Samples may be stored up to 28 days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.

Accuracy Check

Standard Additions Method- Powder Pillows

a) Snap the neck off a Sulfate Standard PourRite Ampule, 1000 mg/L SO_4^{2-} .

b) Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to the three 10-mL samples. Mix thoroughly.

c) Analyze each sample as described above. The sulfate concentration should increase 10 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Additions Method- AccuVac Ampuls

a) Snap the neck off a Sulfate Standard PourRite Ampule, 2500 mg/L SO_4^{2-} .

b) Fill three 25- mL graduated cylinders with 25 mL of sample. Use a TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard to the three cylinders. Mix thoroughly. For AccuVac Ampuls, transfer to a 50-mL beaker.

c) Analyze each sample as described above. The sulfate concentration should increase 10 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Check the accuracy of the test by using the Sulfate Standard Solution, 50 mg/L, listed under Optional Reagents. Or, prepare this solution by pipetting 1.0 mL of a PourRite Ampule Standard for Sulfate (2500 mg/L) into a 50-mL volumetric flask. Dilute to volume with deionized water. The final concentration is 50 mg/L sulfate. Substitute this standard for the sample and proceed with the test as described in the procedure.

Standard Adjust
Standard adjust is recommended when using stored programs 91 or 92. It **should not** be used with a user-entered calibration.

To adjust the calibration curve using the reading obtained with the
50-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **50** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustmen*t for more information.

Method Performance

Precision
In a single laboratory, using a standard solution of 50 mg/L sulfate and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of ±0.5 mg/L sulfate.

In a single laboratory, using a standard solution of 50 mg/L sulfate and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±3 mg/L sulfate.

Estimated Detection Limit (EDL)
The EDL for program 91 is 4.9 mg/L SO_4 and the EDL for program 92 is 3 mg/L SO_4. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

The following interfere at levels above those concentrations listed:

Calcium: 20,000 mg/L as $CaCO_3$	Magnesium: 10,000 mg/L as $CaCO_3$
Chloride: 40,000 mg/L as Cl^-	Silica: 500 mg/L as $CaCO_3$

Summary of Method

Sulfate ions in the sample react with barium in the SulfaVer 4 Sulfate Reagent to form insoluble barium sulfate. The amount of turbidity formed is proportional to the sulfate concentration. The SulfaVer 4 also contains a stabilizing agent to hold the precipitate in suspension.

REQUIRED REAGENTS AND APPARATUS (Using Powder Pillows)

Description	Quantity Required Per Test	Units	Cat. No.
SulfaVer 4 Sulfate Reagent Powder Pillows	1 pillow	100/pkg	21067-69
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

REQUIRED REAGENTS AND APPARATUS (Using AccuVac Ampuls)

SulfaVer 4 Sulfate AccuVac Ampuls	1 ampul	25/pkg	25090-25
Beaker, 50-mL	1	each	500-41H

OPTIONAL REAGENTS

Standard, Drinking Water Inorganics, F^-, NO_3^{-N}, PO_4^{-3}, SO_4^{-2}		500 mL	28330-49
Standard, Wastewater Effluent Inorganics, NH_3^{-N}, NO_3^{-N}, PO_4^{-3}, COD, SO_4^{-2}, TOC		500 mL	28332-49
Sulfate Standard Solution, 50 mg/L		500 mL	2578-49
Sulfate Standard Solution, 1000 mg/L		500 mL	21757-49
Sulfate Standard Solution, PourRite Ampule, 2500 mg/L, 10 mL		16/pkg	14252-10
Sulfate Standard Solution, PourRite Ampule, 1000 mg/L, 2 mL		20/pkg	21757-20
Water, deionized		4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit		each	24052-00
Cylinder, graduated mixing, 25 mL		each	20886-40
Filter Paper, folded, 12.5 cm		100/pkg	1894-57
Flask, volumetric, 50 mL, Class A		each	14574-41
Funnel, poly, 65 mm		each	1083-67
Pipet, TenSette, 0.1 to 1.0 mL		each	19700-01
Pipet Tips, for 19700-01 Pipet		50/pkg	21856-96
Pipet, volumetric, 1.00 mL, Class A		each	14515-35
Pipet, volumetric, 2.00 mL, Class A		each	14515-36
Pipet, volumetric, 3.00 mL, Class A		each	14515-03
Pipet, volumetric, 4.00 mL, Class A		each	14515-04
Pipet, volumetric, 5.00 mL, Class A		each	14515-37
Pipet, volumetric, 6.00 mL, Class A		each	14515-06
Pipet, volumetric, 7.00 mL, Class A		each	14515-07
Pipet Filler, safety bulb		each	14651-00
PourRite Ampule Breaker		each	24846-00

For Technical Assistance, Price and Ordering

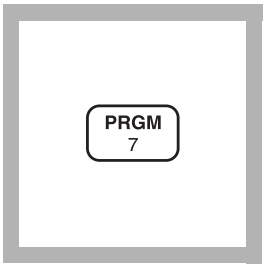
In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

SULFIDE (0 to 0.70 mg/L S^{2-})

For water, wastewater and seawater

Methylene Blue Method* USEPA accepted for reporting wastewater analysis**

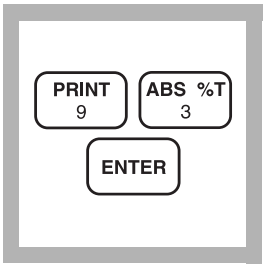


1. Enter the stored program number for sulfide (S).

Press: **PRGM**

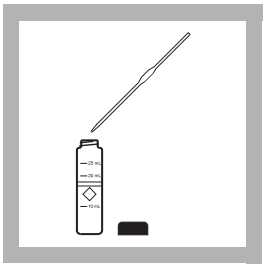
The display will show:

PRGM ?



2. Press: **93 ENTER**

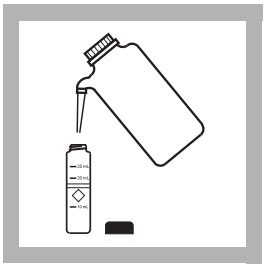
The display will show **mg/L, S** and the **ZERO** icon**.**



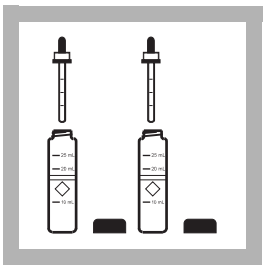
3. Pipet 25 mL of sample into a clean sample cell. This will be the prepared sample.

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis. Use a pipet to avoid agitation.*

Note: *For field testing, a 25-mL graduated cylinder may be used.*

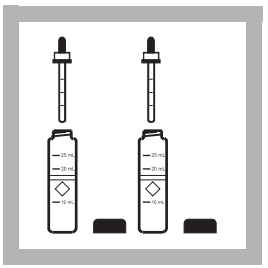


4. Fill a second sample cell with 25 mL of deionized water (the blank).



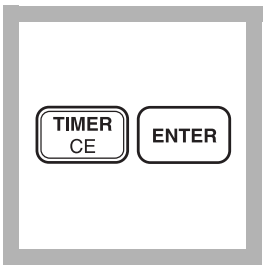
5. Add 1.0 mL of Sulfide 1 Reagent to each cell. Swirl to mix.

Note: *Use the calibrated 1-mL dropper.*



6. Add 1.0 mL of Sulfide 2 Reagent to each cell. Immediately swirl to mix.

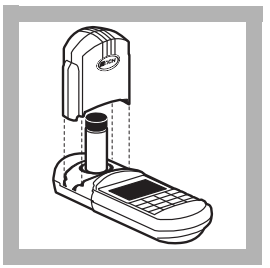
Note: *A pink color will develop, then the solution will turn blue if sulfide is present.*



7. Press:

TIMER ENTER

A 5-minute reaction period will begin.

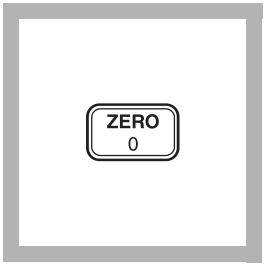


8. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

---

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

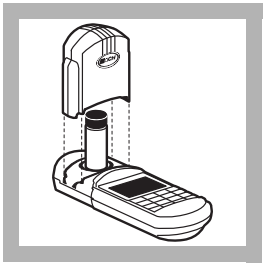
** Procedure is equivalent to USEPA method 376.2 or Standard Method 4500-S^{2-} D for wastewater.



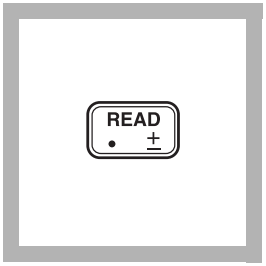
9. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.00 mg/L S



10. After the timer beeps, place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L sulfide will be displayed.

Note: *Some sulfide loss may occur if dilution is necessary.*

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation or prolonged exposure to air. Analyze samples immediately.

Accuracy Check

Sulfide standards are unstable and must be user prepared. See Sandard Methods, 4500S^{-} for preparation and standardization directions.

Method Performance

Precision

In a single laboratory, using standard solutions of 0.73 mg/L sulfide and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ± 0.02 mg/L sulfide.

Estimated Detection Limit (EDL)

The EDL for program 93 is 0.01 mg/L S^{2-}. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Strong reducing substances (sulfite, thiosulfate and hydrosulfite)	Interfere by reducing the blue color or preventing its development.
Sulfide, high levels	High concentrations of sulfide may inhibit full color development and require sample dilution. Some sulfide loss may occur when the sample is diluted.
Turbidity	For turbid samples, prepare a sulfide-free blank as follows. Use it in place of the deionized water blank in the procedure. **1.** Measure 25 mL of sample into a 50-mL erlenmeyer flask. **2.** Add Bromine Water dropwise with constant swirling until a permanent yellow color just appears. **3.** Add Phenol Solution dropwise until the yellow color just disappears. Use this solution in Step 4 in place of deionized water.

Soluble Sulfides

Determine soluble sulfides by centrifuging the sample in completely filled, capped tubes and analyzing the supernatant. Insoluble sulfides are then estimated by subtracting the soluble sulfide concentration from the total sulfide result.

Summary of Method

Hydrogen sulfide and acid-soluble metal sulfides react with N, N-dimethyl-p-phenylenediamine oxalate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. High sulfide levels in oil field waters may be determined after dilution.

Pollution Prevention and Waste Management

Sulfide 2 Reagent contains potassium dichromate. The final solution will contain hexavalent chromium (D007) at a concentration regulated as a hazardous waste by Federal RCRA. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS

	Cat. No.
Sulfide Reagent Set (100 tests)	22445-00
Includes: (2) 1816-42, (2) 1817-42	

Description	Quantity Required Per Test	Units	Cat. No.
Sulfide 1 Reagent	2 mL	100 mL MDB	1816-32
Sulfide 2 Reagent	2 mL	100 mL MDB	1817-32
Water, deionized	25 mL	4 L	272-56

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508-40
Pipet, volumetric, Class A, 25.00 mL	1	each	14515-40
Pipet Filler, safety bulb	1	each	14651-00
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Description	Units	Cat. No.
Bromine Water, 30 g/L	29 mL	2211-20
Phenol Solution, 30 g/L	29 mL	2112-20
Sodium Sulfide, hydrate	114 g	785-14

OPTIONAL APPARATUS

Bottle, Wash, 250 mL	each	620-31
Dropper, for 1 oz. bottle	each	2258-00
Flask, erlenmeyer, 50 mL	each	505-41
Standard Methods for the Examination of Water and Wastewater	each	22708-00

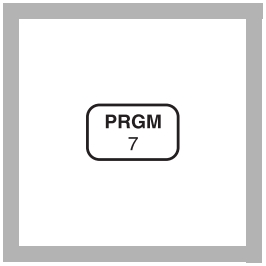
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

SURFACTANTS, ANIONIC (0 to 0.300 mg/L)

(Also called: Detergents) Crystal Violet Method*

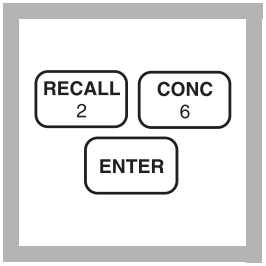


1. Enter the stored program number for Surfactants, anionic (LAS).

Press: **PRGM**

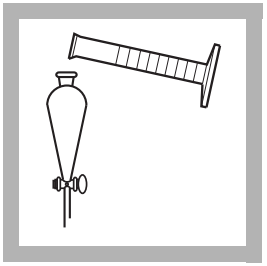
The display will show:

PRGM ?

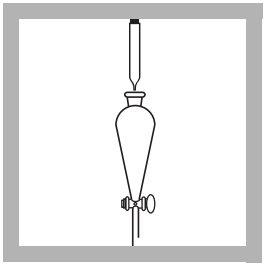


2. Press: **26 ENTER**

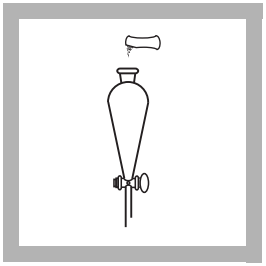
The display will show **mg/L, LAS** and the **ZERO** icon**.**



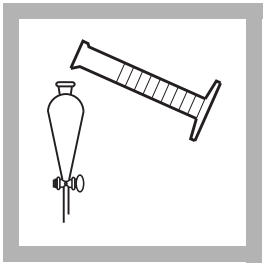
3. Fill a clean 500-mL graduated cylinder to the 300-mL mark with sample. Pour the sample into a clean 500-mL separatory funnel.



4. Add 10 mL of Sulfate Buffer Solution. Stopper the funnel. Shake the funnel for five seconds.



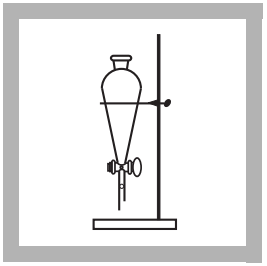
5. Add the contents of one Detergents Reagent Powder Pillow to the funnel. Stopper the funnel and shake to dissolve the powder.



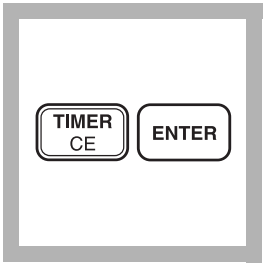
6. Add 30 mL of benzene to the funnel. Stopper the funnel and shake gently for one minute.

Note: *Spilled reagent will affect test accuracy and is hazardous to the skin and other materials.*

Note: *Use benzene only in a well-ventilated area.*



7. Place the separatory funnel in a support stand.



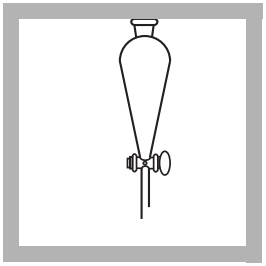
8. Press:

TIMER ENTER

A 30-minute reaction period will begin.

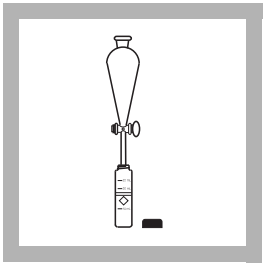
Note: *Excessive agitation may cause an emulsion, requiring a longer time for phase separation. If this occurs, remove most of the water layer, then gently agitate the funnel with a clean inert object in the funnel such as a Teflon-coated magnetic stirring bar.*

* *Analytical Chemistry*, 38, 791(1966).



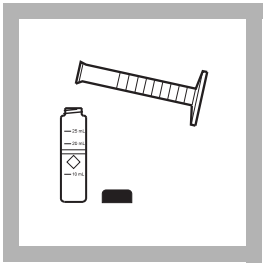
9. After the timer beeps, remove the stopper and drain the bottom water layer. Discard this layer.

Note: *Benzene solutions are a regulated waste and cannot be poured down the drain. See Section 3 for proper disposal of these materials.*

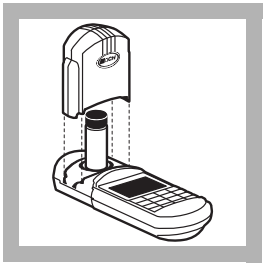


10. Drain the top benzene layer into a clean sample cell (the prepared sample).

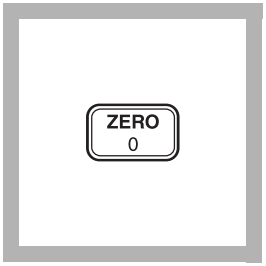
Note: *The benzene layer cannot be filtered before color measurement. Filtration removes the blue color.*



11. Fill another sample cell to the 25-mL mark with pure benzene (the blank).



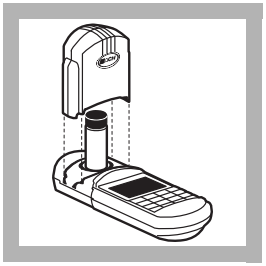
12. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



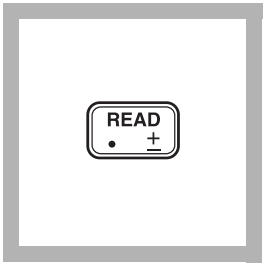
13. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.000 mg/L LAS



14. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



15. Press: **READ**

The cursor will move to the right, then the result in mg/L anionic surfactants (LAS) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Note: *Acetone may be used to clean benzene from glassware.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible, but they may be stored at least 24 hours by cooling to 4 °C
(39 °F). Warm to room temperature before testing.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Detergent Voluette Ampule Standard Solution, 60 mg/L as LAS (The molecular weight of linear alkylate sulfonate used to make the standard is 342).

b) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard to three 300-mL samples. Mix thoroughly.

c) Analyze each as described above. The anionic surfactants reading should increase 0.02 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* (Section 1) for more information.

Method Performance

Precision

In a single laboratory, using a standard solution of 0.150 mg/L LAS, two lots of reagent, and the instrument, a single operator obtained a standard deviation of ±0.010 mg/L LAS as anionic surfactant.

Estimated Detection Limit

The estimated detection limit for program 26 is 0.020 mg/L LAS. For more information on the estimated detection limit, see *Section 1*.

Interferences

Perchlorate and periodate ions will interfere. High amounts of chloride, such as those levels found in brines and seawater, will cause low results.

Summary of Method

Detergents, ABS (alkyl benzene sulfonate) or LAS (linear alkylate sulfonate) are determined by association with crystal violet dye and extraction of the ion-pair complex into benzene.

Pollution Prevention and Waste Management

Benzene (D018) solutions are regulated as hazardous waste by Federal RCRA. Do not pour these materials down the drain. Collect water saturated with benzene solutions for disposal with laboratory solvent wastes. See *Section 3* for more information on proper disposal of these materials.

REQUIRED REAGENTS

Description	Quantity Required Per Test	Unit	Cat. No.
Benzene, ACS	55 mL	500 mL	14440-49
Buffer Solution, sulfate type	10 mL	500 mL	452-49
Detergent Reagent Powder Pillow	1 pillow	25/pkg	1008-68

REQUIRED APPARATUS

Clippers, for opening powder pillows	1	each	968-00
Cylinder, graduated, 25 mL	1	each	508-40
Cylinder, graduated, 50 mL	1	each	508-41
Cylinder, graduated, 500 mL	1	each	508-49
Funnel, separatory, 500 mL	1	each	520-49
Ring, support, 4 inch	1	each	580-01
Sample Cell, 10-20-25 mL, w/ cap	2	6/pkg	24019-06
Stand, support, 127 x 203 mm (5 x 8")	1	each	563-00

OPTIONAL REAGENTS

Acetone, ACS	500 mL	14429-49
Detergent Standard Solution, Voluette ampule, 60 mg/L as LAS, 10 mL	16/pkg	14271-10

OPTIONAL APPARATUS

Ampule Breaker Kit	each	21968-00
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 Pipet	1000/pkg	21856-28
Thermometer, -20 to 110 °C, Non-Mercury	each	26357-02

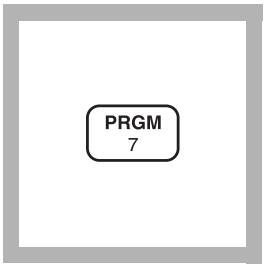
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

SUSPENDED SOLIDS (0 to 750 mg/L)

Photometric Method* (Also called Nonfilterable Residue)

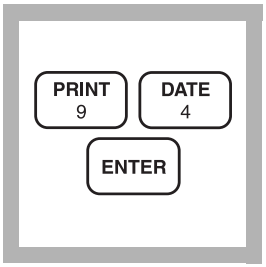


1. Enter the stored program number for suspended solids.

Press: **PRGM**

The display will show:

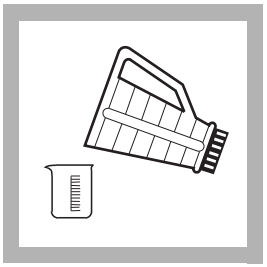
PRGM ?



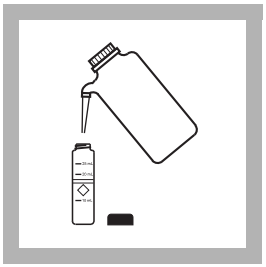
2. Press: **94 ENTER**

The display will show **mg/L, SuSld** and the **ZERO** icon**.**

3. Blend 500 mL of sample in a blender at high speed for exactly 2 minutes.

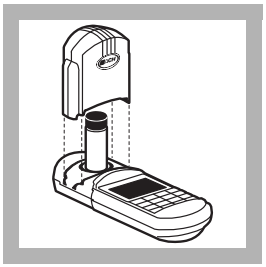


4. Pour the blended sample into a 600-mL beaker.

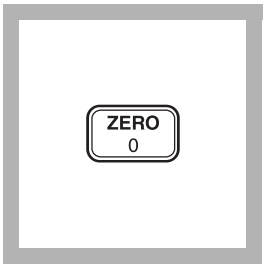


5. Fill a sample cell with 25 mL of tap water or deionized water (the blank).

Note: *Remove gas bubbles in the water by swirling or tapping the bottom of the cell on a table.*



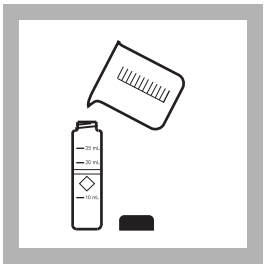
6. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.



7. Press: **ZERO**

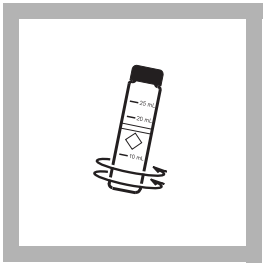
The cursor will move to the right, then the display will show:

0 mg/L SuSld

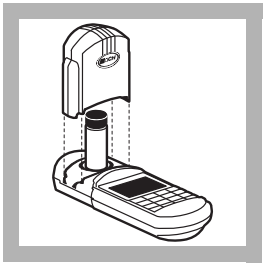


8. Stir the sample thoroughly and immediately pour 25 mL of the blended sample into a sample cell (the prepared sample).

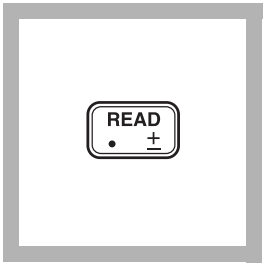
* Adapted from *Sewage and Industrial Wastes*, 31, 1159 (1959).



9. Swirl the prepared sample cell to remove any gas bubbles and uniformly suspend any residue.



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L suspended solids will be displayed.

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible after collection. The sample may be stored seven days by cooling to 4 °C (39 °F).

Method Performance

Precision

In a single laboratory, using a standard solution of 847.4 mg/L Suspended Solids with the instrument, a single operator obtained a standard deviation of ±18.2 mg/L Suspended Solids.

For more information on Hach's precision statement, see *Section 1*.

Estimated Detection Limit

The estimated detection limit for program 94 is 22.1 mg/L Suspended Solids. For more information on the estimated detection limit, see *Section 1*.

Interferences

Calibration for this test is based on parallel samples using the gravimetric technique on sewage samples from a municipal sewage plant. For most samples, this calibration will provide satisfactory results. When higher accuracy is required, run parallel photometric and gravimetric determinations with portions of the same sample. The new calibration should be made on your particular sample using a gravimetric technique as a basis.

Summary of Method

This method of determining suspended solids is a simple, direct measurement which does not require the filtration or ignition and weighing steps that gravimetric procedures do. The USEPA specifies the gravimetric method for solids determinations, while this method is often used for checking in-plant processes.

REQUIRED APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Beaker, 600 mL, low form	1	each	1080-52
Blender, 1.2 L, 120 V	each	each	26161-00
Blender, 1.2 L, 240 V	each	each	26161-02
Cylinder, graduated, 500 mL, poly	1	each	1081-49
Pipet, serologic, 25 mL	1	each	2066-40
Pipet, Filler, safety bulb	1	each	14651-00

OPTIONAL APPARATUS

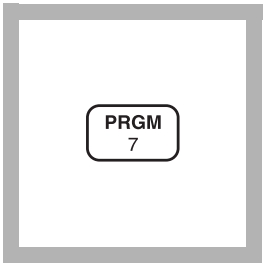
Stirring Rod, glass		3/pkg	1770-01
Wash Bottle, 250 mL		each	620-31

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

Tyrosine Method*

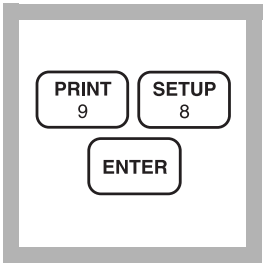


1. Enter the stored program number for tannin and lignin.

Press: **PRGM**

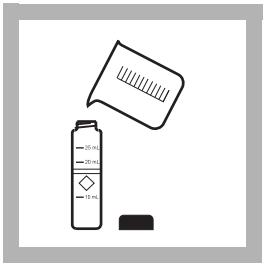
The display will show:

PRGM ?

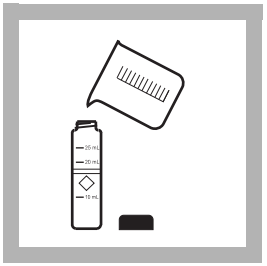


2. Press: **98 ENTER**

The display will show **mg/L, tanic** and the **ZERO** icon**.**

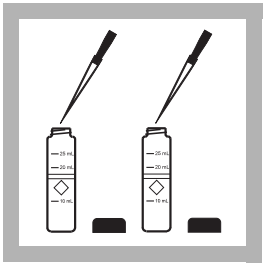


3. Fill a clean sample cell to the 25-mL mark with deionized water (the blank).

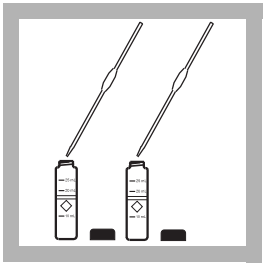


4. Fill a clean sample cell to the 25-mL mark with sample (the prepared sample).

Note: *Filter turbid samples and report results as mg/L soluble tannic acid.*

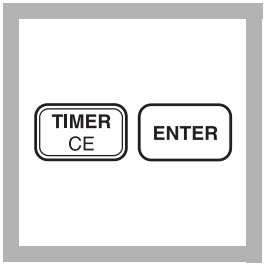


5. Pipet 0.5 mL of TanniVer 3 Tannin-Lignin Reagent into each cell. Swirl to mix.



6. Pipet 5.0 mL of Sodium Carbonate Solution into each cell. Swirl to mix.

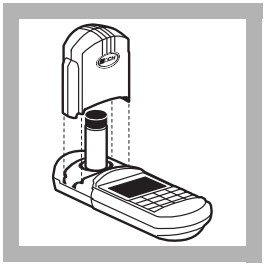
Note: *A blue color will develop if tannins and/or lignins are present.*



7. Press:

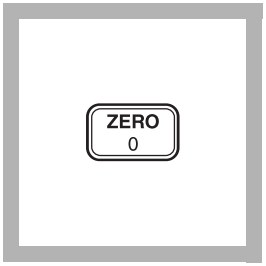
TIMER ENTER

A 25-minute reaction period will begin.



8. Place the blank in the cell holder. Tightly cover the sample cell with the instrument cap.

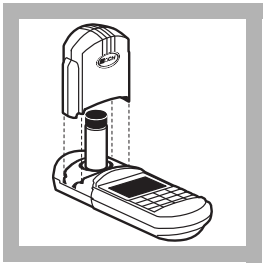
* Adapted from Kloster, M.B., *Journal American Water Works Association,* Vol. 66, No. 1, p. 44 (1974).



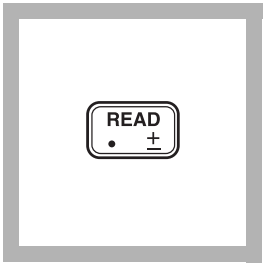
9. Press: **ZERO**

The cursor will move to the right, then the display will show:

0.0 mg/L tanic



10. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L tannic acid will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Standard Adjust in Section 1).*

Sampling and Storage

Collect samples in clean plastic or glass bottles.

Accuracy Check

Standard Solution Method

Prepare a 200-mg/L tannic acid standard solution by dissolving 0.200 grams of tannic acid in deionized water and diluting to 1000 mL. Prepare this solution monthly. A 2.0 mg/L tannic acid standard is prepared by diluting 10.00 mL of the stock solution to 1000 mL with deionized water. Prepare this standard daily.

Method Performance

Precision

In a single laboratory, using standard solutions of 4.0 mg/L tannic acid and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.1 mg/L tannic acid.

Estimated Detection Limit

The estimated detection limit for program 98 is 0.1 mg/L tannin and lignin. For more information on the estimated detection limit, see *Section 1*.

Interferences

Substance	Interference Level and Treatment
Ferrous iron	Causes a positive interference. Two mg/L of ferrous iron produces a color equivalent to about 1 mg/L of tannic acid. To eliminate interference of ferrous iron up to 20 mg/L, add one 0.2-g scoop of sodium pyrophosphate to the sample before testing.
Sulfide	Eliminated by adding 1 mL of formaldehyde to the sample before testing the sample.

Summary of Method

This test measures all hydroxylated aromatic compounds, including tannin, lignin, phenol and cresol. This method produces a blue color proportional to the amount of these compounds present in the sample. Report results as total tannin and lignin expressed as mg/L tannic acid.

REQUIRED REAGENTS

Cat. No.

Tannin and Lignin Reagent Set (up to 100 tests) 22446-00
Includes: (2) 675-49, (1) 2560-42

Description	Quantity Required Per Test	Unit	Cat. No
Sodium Carbonate Solution	10 mL	500 mL	675-49
TanniVer 3 Tannin-Lignin Reagent	1 mL	100 mL	2560-42
Water, deionized	25 mL	4 L	272-56

REQUIRED APPARATUS

Pipet Filler, safety bulb	1	each	14651-00
Pipet, volumetric, Class A, 5.0 mL	1	each	14515-37
Pipet, volumetric, Class A, 0.5 mL	1	each	14515-34
Sample Cell, 10-20-25-mL, w/ cap	2	6/pkg	24019-06

OPTIONAL REAGENTS

Formaldehyde	100 mL	2059-32
Sodium Pyrophosphate, ACS	50 g	14295-25
Tannic Acid	113 g	791-14

OPTIONAL APPARATUS

Description	Unit	Cat. No
Balance, analytical, 115 V	each	28014-01
Balance, analytical, 230 V	each	28014-02
Cylinder, graduated, 25 mL	each	508-40
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, volumetric, 1000 mL	each	14547-53
Funnel, poly, 65 mm	each	1083-67
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Pipet, Filler, safety bulb	each	14651-00
Spoon, measuring, 0.2 g	each	638-00
Weighing Boat, 67/47 mm	500/pkg	21790-00

For Technical Assistance, Price and Ordering

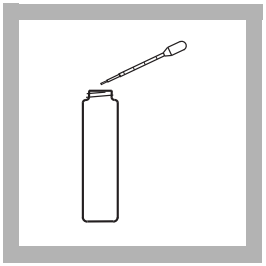
In the U.S.A.—Call 800-227-4224

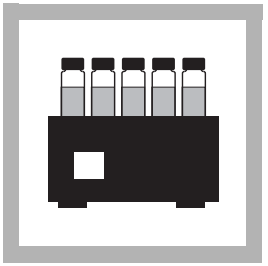
Outside the U.S.A.—Contact the Hach office or distributor serving you.

TOXTRAK™ TOXICITY TEST*

Colorimetric Method**

Inoculum Development



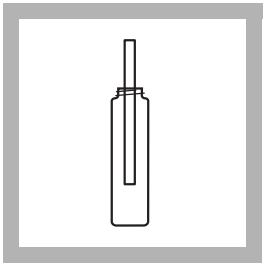


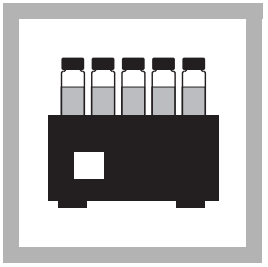
Using Indigenous Biomass

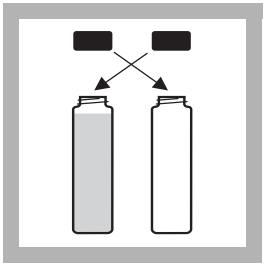
1. Using one of the dropper pipets provided, add 1.0 mL of source culture to a Tryptic Soy Broth Tube.

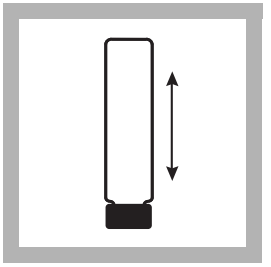
2. Incubate at 37 °C until the vial contents are visibly turbid (turbidity indicates bacterial growth).

Inoculum Development Using Aqua QC-Stiks









1. Inoculate a Lauryl Tryptose broth tube with an *E. coli* Aqua QC-Stik™ according to the instructions that come with the stick.

2. Incubate the Lauryl Tryptose Broth Tube at 35°C (95°F) until the medium is visibly turbid (approxamately 12 hours). Turbidity develops much faster in an incubator than at room temperature.

3. Inoculate a new Lauryl Tryptose Broth Tube by first inverting the tube and switching the caps of the two tubes.

In this way, several medium vials can be inoculated from one Aqua–QC Stick™.

4. Invert the new tube. After incubation, this new vial may be used in subsequent tests.

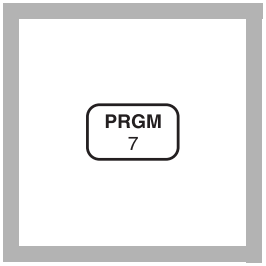
If toxicity tests will be run on consecutive days, inoculum may be kept several days at room temperature.

Cultures 10 to 72 hours old give best results.

* U.S. Patent number 5,413,916.

** Liu, D., *Bull. Environ. Contam. Toxicol.* 26, 145-149 (1981).

Colorimetric Reaction

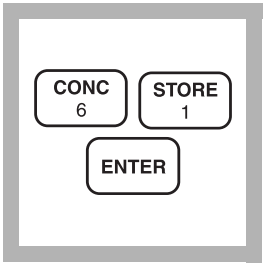


1. Enter the stored program number for toxicity.

Press: **PRGM**

The display will show:

PRGM ?

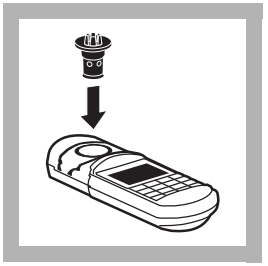


2. Press: **61 ENTER**

The display will show:

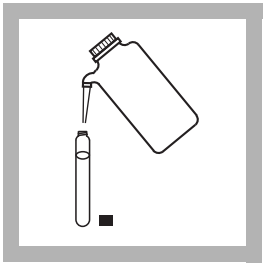
ABS 610 nm

and the zero icon.

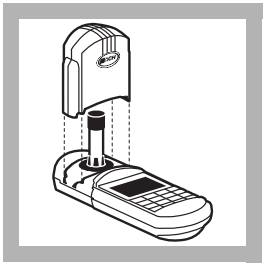


3. Insert the TNT/COD Adapter into the cell holder by rotating the adapter until it drops into place. Then push down to fully insert it.

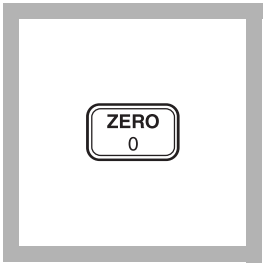
Note: *A diffuser band covers the light path holes on the adapter to give increased performance. The band should NOT be removed.*



4. Fill a Test 'N Tube vial with deionized water. Label this vial as the "blank". Wipe the outside of all the vials with a tissue to remove fingerprints and smudges.



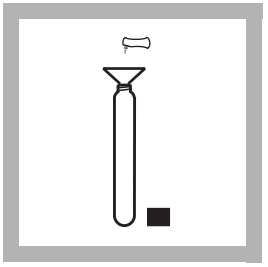
5. Place the blank in the adapter. Tightly cover the vial with the instrument cap.



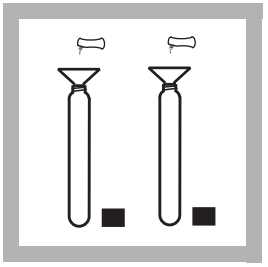
6. Press: **ZERO**

The cursor will move to the right, then the display will show:

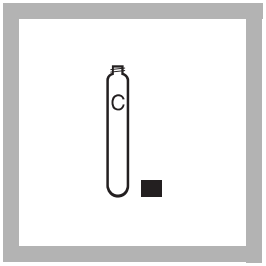
0.000 ABS



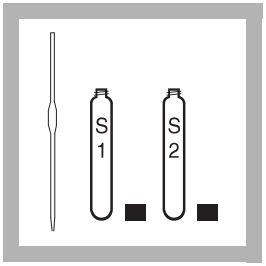
7. Label a vial "control." Open one ToxTrak Reagent Powder Pillow and add the contents to the empty reaction vial.



8. Label each sample or dilution vial clearly. Add the contents of one ToxTrak Reagent Powder Pillow to each labeled vial.

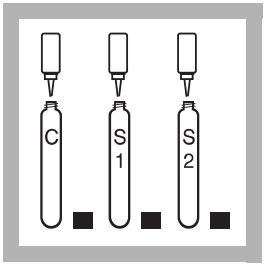


9. Add 5.0 mL of deionized water to the control tube.

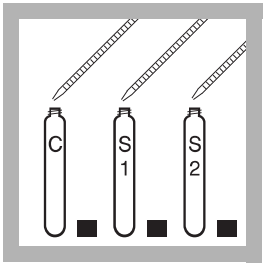


10. Add 5.0 mL of the sample (or dilution) to each sample vial.

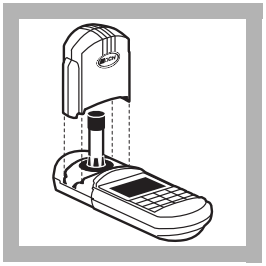
Note: *To determine the approximate threshold level of toxicity for a sample, dilute 1 mL of sample to 10 mL of deionized water and run the test. Continue to make serial 1:10 dilutions until a level is reached which gives a 0% Inhibition in Step 18.*



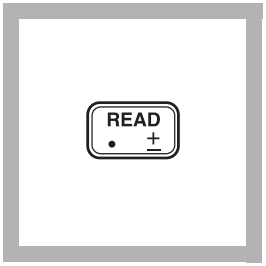
11. Add 2 drops of Accelerator Solution to each vial. Cap and invert to mix.



12. Add 0.5 mL of inoculum (previously prepared) to each vial. Cap and invert to mix.



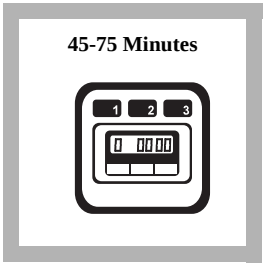
13. Place the control vial in the cell holder. Tightly cover the vial with the instrument cap.



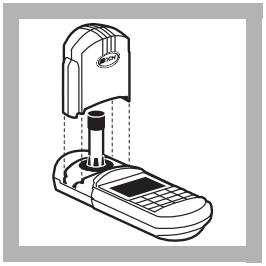
14. Press: **READ**

The cursor will move to the right, then the result in ABS will be displayed. Record the absorbance of the "control" vial.

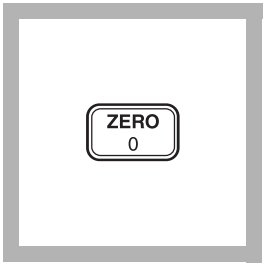
15. Repeat Steps 13-14 for all samples and dilutions. Be sure to record each absorbance.



16. Allow the solutions in the tubes to react until the absorbance of the **control tube** decreases 0.60 ±0.10.
This should take about 45-75 minutes.



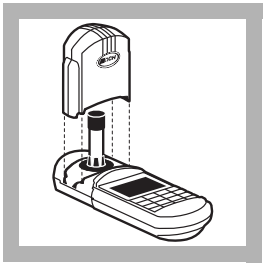
17. After the absorbance of the "control" vial has decreased 0.60 ±0.10 absorbance units, place the blank in the adapter. Tightly cover the vial with the instrument cap.



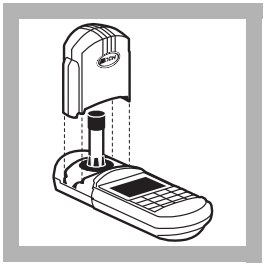
18. Press: **ZERO**

The cursor will move to the right, then the display will show:

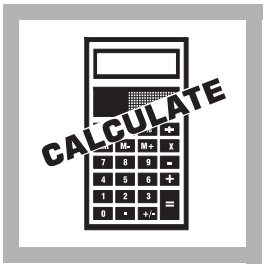
0.000 ABS



19. Place the "control" vial in the cell holder. Tightly cover the vial with the instrument cap. Record the absorbance value of the control.



20. Place each sample or dilution vial in the cell holder. Tightly cover the vial with the instrument cap. Record each absorbance value.



21. Calculate the % Inhibition as follows:

$$\%I = \left[1 - \left(\frac{\Delta \text{Abs sample}}{\Delta \text{Abs control}}\right)\right] \times 100$$

ΔA is the initial absorbance value minus the final absorbance value.

See the example following this step.

Note: *Some toxins increase respiration and will give a negative % inhibition on all respiration-based toxicity tests. After repeated testing, samples which always give a % inhibition in Step 21 that is more negative than –10% should be considered toxic.*

Example

The control tube (C) has an initial absorbance of 1.6 and decreases to 1.0 Abs. The sample tube has an initial absorbance of 1.7 and decreases to 1.3 Abs.

ΔAbs. Sample = 1.7 – 1.3 = 0.4 ΔAbs. Control = 1.6 – 1.0 = 0.6

$$\%I = \left(1 - \left(\frac{0.4}{0.6}\right)\right) \times 100 \qquad \%I = 33.3$$

Interpreting Results

The Percent Inhibition results obtained are only a relative measurement. They do not represent a true quantitative measurement of toxic concentration. The Percent Inhibition does not necessarily increase in direct proportion to the concentration of toxins. To determine the minimum inhibition concentration of a toxin, it is possible to make tenfold dilutions of the sample and determine the Percent Inhibition for the dilutions. When the sample is diluted so that no inhibition is observed, this is the No Observed Effect Concentration (NOEC).

Due to the many variables involved in the test, the limits of detection are on the order of 10% Inhibition. This would correlate to the Lowest Observable Effect Concentration (LOEC). If a sample shows less that 10% Inhibition, repeat the test. After several repetitions, look at the series of data to determine the likelihood of toxicity. Results below 10% are not reliable, but can be used to surmise some presence of toxicity if they are consistent. See the table below.

Toxicity Results

Data Points: Percent Inhibition	Conclusion
7%, 9%, 5%, 8%, 5%	May be slightly toxic
7%, –4%, 5%, 5%, 1%	Most likely not toxic
–7%, –9%, 5%, –8%, –5%	May be slightly toxic

Some toxins will increase respiration and will give a negative Percent Inhibition on this and all other respiration-based toxicity tests. After repeated testing, samples that always give a Percent Inhibition that is more negative than –10% should be considered toxic.

Disposal of Test Cultures

Dispose of active bacterial cultures by using one of these methods:

1. Autoclave used test containers at 121 °C for 15 minutes at 15 pounds of pressure. Once the containers are sterile, pour the contents down the drain with running water. The reaction tubes may be washed
and re-used.

2. Sterilize test containers by using a 1:10 dilution of commercial

laundry bleach. Pour the test container contents and test containers into the bleach solution. Allow 10-15 minutes of contact time with the bleach solution. Then pour the liquid down the drain and wash the reaction tubes for re-use.

Summary of Method

Resazurin is a redox-active dye, which changes from pink to blue when it is reduced. Bacterial respiration occurring in the sample reduces resazurin. If toxic substances are present, they inhibit the rate of resazurin reduction. The sample color is compared to a toxin-free control tube to determine how toxic the sample is to an indigenous culture or a culture of *E. coli*. A chemical accelerant reduces the reaction time of the procedure.

REQUIRED REAGENTS

Description	Cat. No.
ToxTrak Reagent Set (25 tests)	25972-00
Includes: (1) 25607-66, (1) 25608-36, (1) 22777-00, (1) 24092-32	

Description	Quantity Required Per Test	Unit	Cat. No.
Aqua QC–Stiks, Escherichia Coli	1	3 cultures	27063-03
Sodium Thiosulfate Standard Solution	varies	100 mL	24092-32
ToxTrak Reagent Powder Pillows	1 pillow	50/pkg	25607-66
ToxTrak Accelerator Solution	2 drops	15 mL SCDB	25608-36
Tryptic Soy Broth Tubes	1	15/pkg	22777-00
Tube, culture, with cap	1	10/pkg	20962-08
Water, deionized	varies	200 mL	272-29

REQUIRED APPARATUS

Cap, White	1	6/pkg	22411-06
Clippers, to open powder pillows	1	each	936-00
COD/TNT Adapter	1	each	48464-00
Dropper Pipet, 1 mL	varies	10/pkg	21247-20
Forceps, flat square tip	1	each	14537-00
Pipet, volumetric, 5.00 mL, Class A	1	each	14515-37
Pipet Filler, Safety Bulb	1	each	14651-00
Vial, Test ‘N Tube	1	6/pkg	25831-25

OPTIONAL APPARATUS

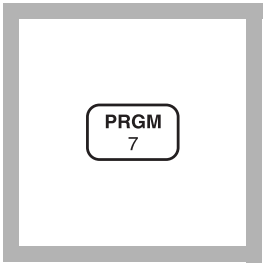
Description	Unit	Cat. No.
Burner, Alcohol, 60 mL	each	20877-42
Burner, Bunsen	each	21627-00
Germicidal Cloth	50/pkg	24632-00
Incubator, Dri Bath, 25 well, 120/230 V	each	45900-00
Incubator, Dri Bath, 25 well, 120/230 V, with European power cord	each	45900-02
Pipet, Sterile Transfer	15/pkg	22325-12
Timer	each	26305-00

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.

TURBIDITY (0 to 1000 FAU)

Absorptometric Method*



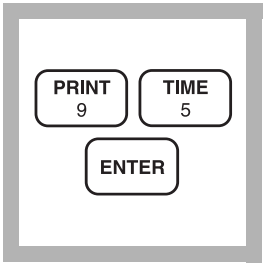
1. Enter the stored program number for turbidity.

Press: **PRGM**

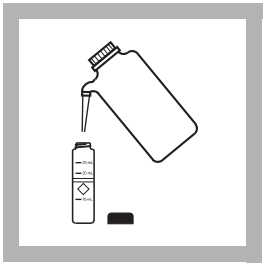
The display will show:

PRGM ?

Note:
1 FAU=1 NTU=1 FTU when measuring formazin. These are not equivalent when measuring other types of standards or samples.



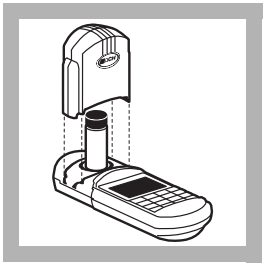
2. Press: **95 ENTER**
The display will show **FAU** and the **ZERO** icon.



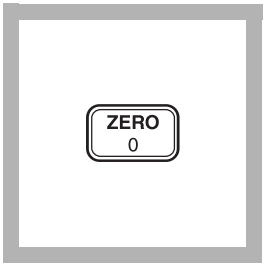
3. Fill a sample cell with 10 mL of deionized water (the blank).

Note: *Wipe the surface of the cell with a soft cloth.*

Note: *For highly colored samples, use a filtered portion of sample in place of the deionized water.*



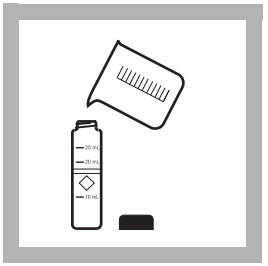
4. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



5. Press: **ZERO**

The cursor will move to the right, then the display will show:

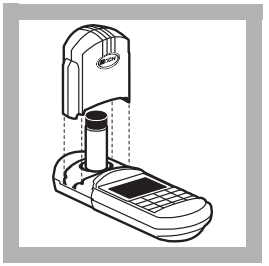
0 FAU



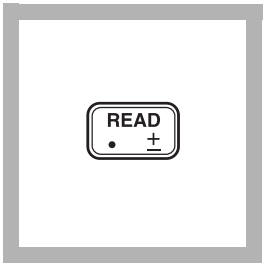
6. Fill another sample cell with 10 mL of sample.

Note: *Mix the sample well before transferring it to the sample cell.*

Note: *Wipe the surface of the cell with a soft cloth.*



7. Place the sample cell into the cell holder. Tightly cover the sample cell with the instrument cap.



8. Press: **READ**

The cursor will move to the right, then the result in Formazin Attenuation Units (FAU) will be displayed.

Note: *Standard Adjust may be performed using a prepared standard (see Section I).*

* Adapted from FWPCA *Methods for Chemical Analysis of Water and Wastes*, 275 (1969)

Sampling and Storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible. Store samples up to 48 hours by cooling to 4 °C (39 °F). Analyze the sample at the same temperature as it was collected.

Accuracy Check

Standard Solution Method

The stored program has been calibrated using formazin, the primary standard for turbidity. A 200 FAU formazin solution for checking the accuracy of the test can be prepared using the following procedure.

1. Pipet 5.00 mL of a 4000 NTU Formazin stock solution into a 100-mL volumetric flask.

2. Dilute to the mark with deionized water. Prepare this daily.

Convenient stabilized turbidity stock solution (200 NTU StablCal™ Standard) is available from Hach.

Standard Adjust

To adjust the calibration curve using the reading obtained with the
200 FAU formazin standard, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **200** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

Method Precision

Precision

In a single laboratory, using a turbidity standard solution of 200 FAU with the instrument, a single operator obtained a standard deviation of ±2 FAU.

Estimated Detection Limit

The estimated detection limit for program 95 is 21 FAU. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Air Bubbles	Interfere at all levels. Degass samples using the Degassing Kit or an ultrasonic bath.
Color	Interferes if the color absorbs light at 520 nm.
Temperature extremes	May interfere by changing the turbidity of the sample. Analyze samples as soon as possible after collection. Analyze at the same temperature as the original sample.

Summary of Method

This turbidity test measures an optical property of the sample which results from scattering and absorption of light by particles in the sample. The amount of turbidity measured depends on variables such as the size, shape, color, and refractive properties of the particles.

This procedure is calibrated using formazin turbidity standards and the readings are in terms of Formazin Attenuation Units (FAU). This test cannot be used for USEPA reporting purposes, but it may be used for daily in-plant monitoring. One FAU is equivalent to one Nephelometric Turbidity Unit (NTU) of Formazin. However, the optical method of measurement for FAU is very different than the NTU method (1 NTU = 1 FTU = 1 FAU when traced to formazin primary standards.)

REQUIRED APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06

REQUIRED REAGENTS

Description	Units	Cat. No.
Formazin Stock Solution, 4000 NTU	500 mL	2461-49
Silicone Oil	15 mL DB	1269-36
StablCal Stabilized Turbidity Standard, 200 NTU	500 mL	26604-49
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Description	Units	Cat. No.
Bath, ultrasonic	each	24895-00
Bottle, wash, 250 mL	each	620-31
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, filter, 500 mL	each	546-49
Filter Holder	each	13529-00
Filter Pump, aspirator	each	2131-00
Oiling cloth, for applying silicone oil	each	26873-00
Pipet Filler, safety bulb	each	14651-00
Pipet, volumetric, Class A, 5.0 mL	each	14515-37
Sample Degassing Kit	each	43975-00
Stopper, rubber, one-hole, No. 7	6/pkg	2119-07
Tubing, rubber, 5/16” I.D.	12 feet	560-19
Tweezers, plastic	each	14282-00

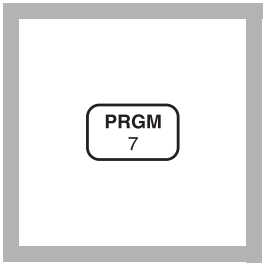
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

VOLATILE ACIDS (0 to 2800 as mg/L HOAc)

Esterification Method*



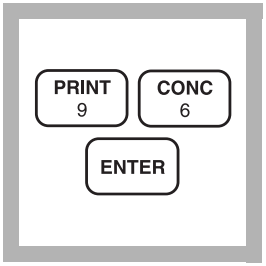
1. Enter the stored program number for Volatile Acids as acetic acid (HOAc).

Press: **PRGM**

The display will show:

PRGM ?

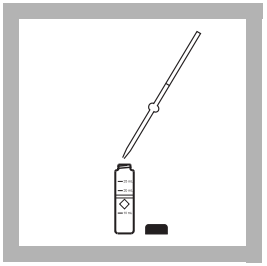
Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **96 ENTER**

The display will show **mg/L, HOAc** and the **ZERO** icon**.**

Note: *If high levels of dissolved solids or mineral acids are present, distill as described in the Hach Distillation Apparatus manual.*



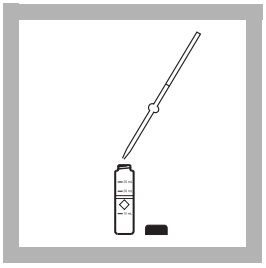
3. Pipet 0.5 mL of deionized water into a dry 25-mL sample cell (the blank).

Note: *Use a Class A or TenSette Pipet.*

Note: *Adjust the pH of stored samples before analysis.*

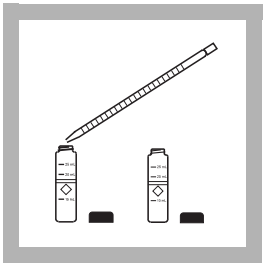
4. Filter or centrifuge 25 mL of the sample.

Note: *Centrifugation is faster than filtration.*

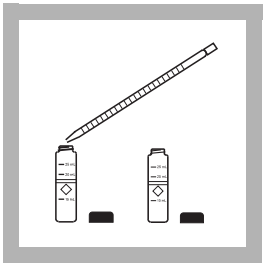


5. Pipet 0.5 mL of the filtrate or supernatant into another dry 25-mL sample cell (the prepared sample).

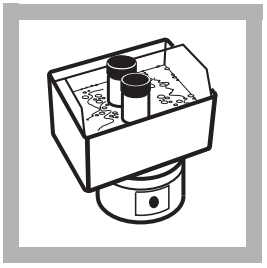
Note: *Use a Class A or TenSette Pipet.*



6. Pipet 1.5 mL of ethylene glycol into each sample cell. Swirl to mix.



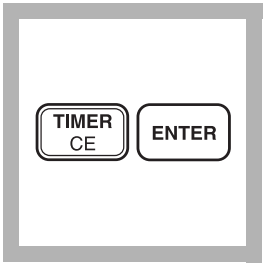
7. Pipet 0.2 mL of 19.2 N Sulfuric Acid Standard Solution into each cell. Swirl to mix.



8. Place both cells into a boiling water bath.

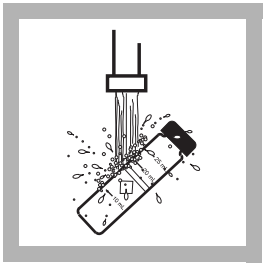
Note: *Samples may be boiled in a 600-mL beaker.*

* Adapted from *The Analyst*, 87, 949 (1962)

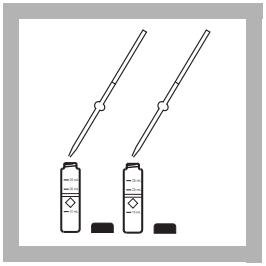


9. Press: **TIMER ENTER**

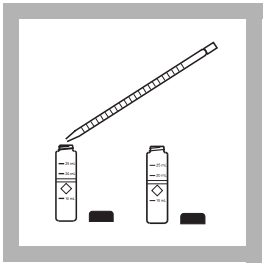
A 3-minute reaction period will begin.



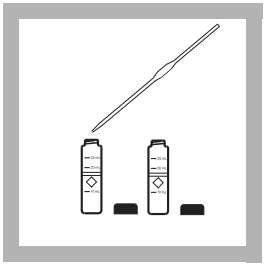
10. When the timer beeps, cool solutions to 25 °C (until cells feel cool) with running tap water. Then dry the cells with a soft cloth.



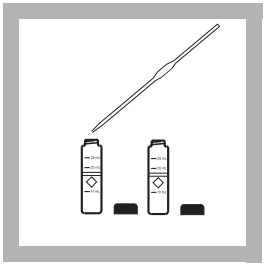
11. Pipet 0.5 mL of Hydroxylamine Hydrochloride Solution into each cell. Swirl to mix.



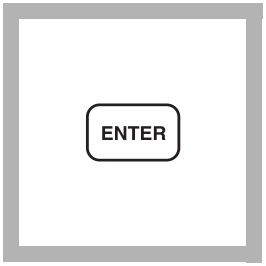
12. Pipet 2.0 mL of 4.5 N Sodium Hydroxide Standard Solution into each cell. Cap and invert to mix.



13. Add 10 mL of Ferric Chloride Sulfuric Acid Solution to each cell. Cap and invert to mix.



14. Add 10 mL of deionized water to each cell. Cap and invert to mix.

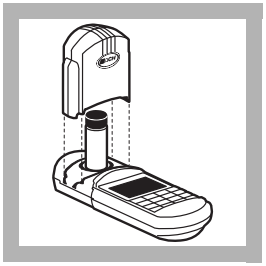


15. The display will show: **3:00 TIMER 2**

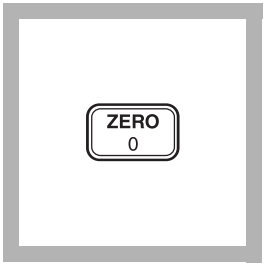
Press: **ENTER**

A 3-minute reaction period will begin.

Note: *After this three-minute reaction period, proceed immediately through steps 16-19.*



16. When the timer beeps, immediately place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

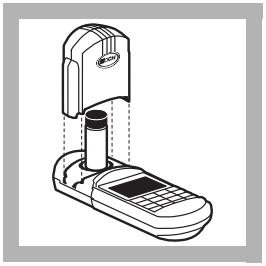


17. Press: **ZERO**

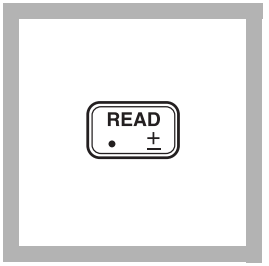
The cursor will move to the right, then the display will show:

0 mg/L HOAc

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



18. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



19. Press: **READ**

The cursor will move to the right, then the result in mg/L Volatile Acids as acetic acid will be displayed.

Sampling and Storage

Collect samples in plastic or glass bottles. Analyze samples as soon as possible after collection. Samples can be stored up to 24 hours by cooling to 4 °C (39 °F) or below. Warm to room temperature before testing.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Volatile Acids PourRite Ampule Standard Solution, 62,500 mg/L as acetic acid.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard, respectively, to three 25-mL graduated mixing cylinders, each containing 25 mL of filtered sample. Stopper. Shake well to mix.

c) Remove a 0.5 mL aliquot of sample from each cylinder; add to three dry sample cells. Analyze all three samples along with the original test sample beginning with Step 5 of the procedure. The volatile acid concentration should increase 250 mg/L volatile acids as acetic acid for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1.*

Standard Solution Method

Prepare a 500 mg/L volatile acid standard by using the TenSette Pipet to add 0.8 mL of a Volatile Acids PourRite Ampule Standard Solution (62,500 mg/L as acetic acid) to a 100-mL volumetric flask. Dilute to volume with deionized water. Stopper and invert to mix.

Method Performance

Precision

In a single laboratory, using a standard solution of 500 mg/L volatile acids as acetic acid and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±8 mg/L.

Estimated Detection Limit

The estimated detection limit for program 96 is 17 mg/L HOAc. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

The volatile acids test is designed specifically for the determination of volatile acids in digestor sludges. The method is based on esterification of the carboxylic acids present and determination of the esters by the ferric hydroxamate reaction. All volatile organic acids present are reported as their equivalent mg/L acetic acid.

REQUIRED REAGENTS

	Cat. No.
Volatile Acids Reagent Set (90 tests)	22447-00
Includes: (1) 2039-53, (2) 2042-53, (1) 818-42, (1) 2040-53, (1) 2038-32	

Description	Quantity Required Per Test	Units	Cat. No.
Ethylene Glycol	3 mL	1000 mL	2039-53
Ferric Chloride-Sulfuric Acid Solution	20 mL	1000 mL	2042-53
Hydroxylamine Hydrochloride Solution, 100 g/L	1 mL	100 mL	818-42
Sodium Hydroxide Standard Solution, 4.5 N	4 mL	1000 mL	2040-53
Sulfuric Acid Standard Solution, 19.2 N	0.4 mL	100 mL	2038-32
Water, deionized	20.5 mL	4 L	272-56

REQUIRED APPARATUS

Description	Quantity Required Per Test	Units	Cat. No.
Cots, finger	2	2/pkg	14647-02
Cylinder, graduated, 10 mL	1	each	508-38
Filter Paper, folded, 12.5 cm	1	100/pkg	1894-57
Flask, erlenmeyer, 50 mL	1	each	505-41
Funnel, poly, 65 mm	1	each	1083-67
Hot Plate, circular, 3.5-inch diameter	1	each	12067-01
Pipet Filler, safety bulb	1	each	14651-00
Pipet, serological, 2 mL	2	each	532-36
Pipet, volumetric, Class A, 0.5 mL	3	each	14515-34
Pipet, volumetric, Class A, 10.00 mL	3	each	14515-38
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
Water Bath and Rack	1	each	1955-55

OPTIONAL REAGENTS

Volatile Acids Standard Solution, PourRite ampule, 62,500 mg/L as acetic acid, 10 mL	16/pkg	14270-10

OPTIONAL APPARATUS

Ampule Breaker, PourRite	each	24846-00
Beaker, 600 mL	each	500-52
Bottle, wash, 500 mL	each	620-11
Centrifuge, laboratory, 115 Vac	each	26765-00
Centrifuge, laboratory, 230 Vac	each	26765-02
Centrifuge Tubes, 15 mL	10/pkg	22787-39
Centrifuge Tube Caps	20/pkg	25852-20
Cylinder, graduated, mixing, 25 mL	each	1896-40
Cylinder, graduated, plastic, 250 mL	each	1081-46
Distillation Apparatus	each	22653-00
Distillation Heater and Support Apparatus	each	22744-00
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet Tips, for 19700-10	50/pkg	21997-96

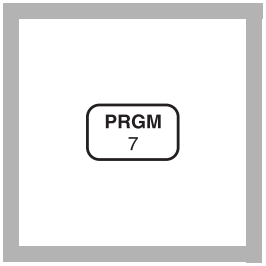
For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

ZINC (0 to 3.00 mg/L Zn)

Zincon Method* USEPA approved for wastewater analysis** (digestion needed; see Section 2)



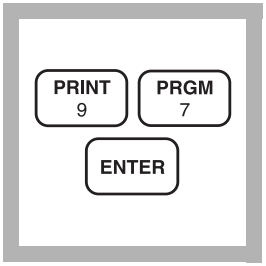
1. Enter the stored program number for zinc (Zn).

Press: **PRGM**

The display will show:

PRGM ?

Note: *For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*

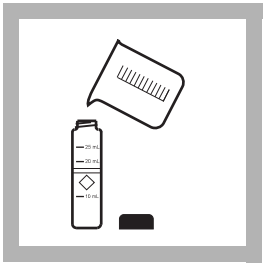


2. Press: **97 ENTER**

The display will show **mg/L, Zn** and the **ZERO** icon**.**

Note: *Total zinc requires a prior digestion; use either the Digesdahl or mild digestion (Section 2).*

Note: *Adjust the sample to pH 4-5; see Sampling and Storage following these steps.*



3. Fill a 25-mL sample cell with 20 mL of sample.

Note: *Rinse glassware with 1:1 hydrochloric acid and deionized water before use.*

Note: *If samples cannot be analyzed immediately, see Sampling and Storage.*

4. Add the contents of one ZincoVer 5 Reagent Powder Pillow. Cap. Invert several times to completely dissolve the powder. If the sample does not turn orange, see the note below.

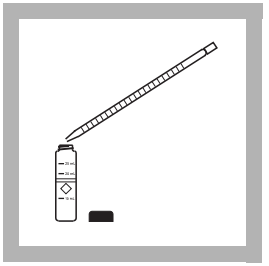
Note: *Powder must be completely dissolved or inconsistent results may occur.*

Note: *The sample should be orange. If it is brown or blue, dilute the sample and repeat the test. Either the zinc concentration is too high or an interference is present.*

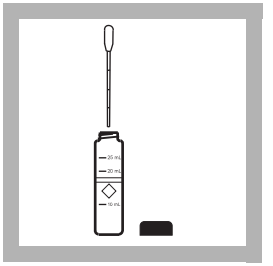
Caution: **ZincoVer 5 contains cyanide and is very poisonous if taken internally or inhaled. Do not add to an acidic sample. Store away from water and acids.**

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*

** *Federal Register,* 45 (105) 36166 (May 29, 1980).

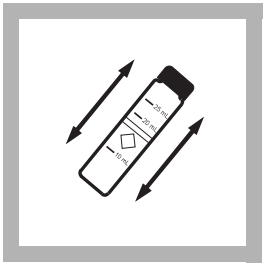


5. Measure 10 mL of the orange solution into another sample cell (the blank).



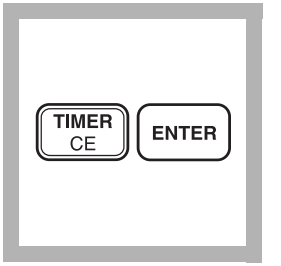
6. Add 0.5 mL of cyclohexanone to the remaining orange solution in the first sample cell (the prepared sample).

Note: *Use a plastic squeezer. Rubber bulbs may contaminate the cyclohexanone.*



7. Tightly cap the cell. Shake vigorously for 30 seconds (the prepared sample).

Note: *The sample will be red-orange, brown or blue, depending on the zinc concentration.*

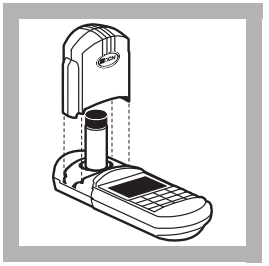


8. Press:

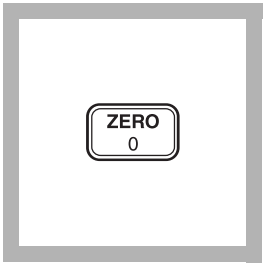
TIMER ENTER

A 3-minute reaction period will begin.

Note: *Steps 9-11 must be completed within 10 minutes after the timer beeps.*



9. Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

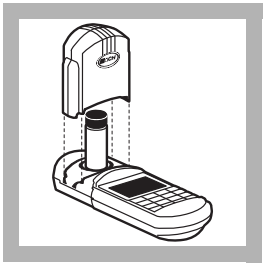


10. Press: **ZERO**

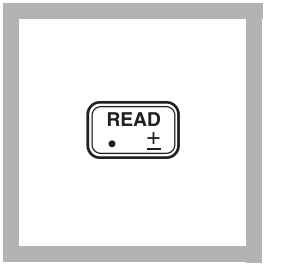
The cursor will move to the right, then the display will show:

0.00 mg/L Zn

Note: *If Reagent Blank Correction is on, the display may flash "limit". See Section 1.*



11. Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**

The cursor will move to the right, then the result in mg/L Zn will be displayed.

Note: *Standard Adjust may be performed using a prepared 0.50 mg/L standard. See Section 1.*

Sampling and Storage

Collect samples in acid-washed plastic bottles. For storage, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). The preserved samples can be stored up to six months at room temperature.

Adjust the pH to 4 to 5 with 5.0 N sodium hydroxide before analysis. Do not exceed pH 5, as zinc may be lost as a precipitate. Correct the test result for volume additions; see *Sampling and Storage, Volume Additions,* in *Section 1* for more information.

If only dissolved zinc is to be determined, filter the sample before the acid addition.

Accuracy Check

Standard Additions Method

a) Snap the neck off a Zinc PourRite Ampule Standard, 25 mg/L Zn.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL samples. Mix each thoroughly.

c) Analyze each sample as described above. The zinc concentration should increase 0.1 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see *Standard Additions* in *Section 1* for more information.

Standard Solution Method

Prepare a 0.50 mg/L zinc standard solution by diluting 5.00 mL of Zinc Standard Solution, 100 mg/L as Zn, to 1000 mL with deionized water in a Class A 1000-mL volumetric flask. Prepare this solution daily. Use this solution as the sample and perform the zinc procedure as described above.

Method Performance

Precision

In a single laboratory, using a standard solution of 1.50 mg/L Zn and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.02 mg/L Zn.

Estimated Detection Limit (EDL)

The EDL for program 97 is 0.02 mg/L Zn. For more information on derivation and use of Hach's estimated detection limit, see *Section 1*.

Interferences

The following may interfere when present in concentrations exceeding those listed below.

Interfering Substance	Interference Level and Treatments
Aluminum	6 mg/L
Cadmium	0.5 mg/L
Copper	5 mg/L
Iron (ferric)	7 mg/L
Manganese	5 mg/L
Nickel	5 mg/L
Organic material	Large amounts may interfere. Perform the mild digestion (Section 2) to eliminate this interference.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment (see pH Interference in Section 1). Adjust pH to 4-5.

Pollution Prevention and Waste Management

ZincoVer 5 reagent contains potassium cyanide. Cyanide solutions are regulated as hazardous wastes by the Federal RCRA. Cyanide should be collected for disposal as reactive (D003) waste. Be sure that cyanide solutions are stored in a caustic solution with pH >11 to prevent the release of hydrogen cyanide gas.

In the event of a spill or release, clean up the area by following these steps:

a) Use a fume hood or supplied-air or self-contained breathing apparatus.

b) While stirring, add the waste to a beaker containing a strong solution of sodium hydroxide and calcium hypochlorite or sodium hypochlorite (household bleach).

c) Maintain a strong excess of hydroxide and hypochlorite. Let the solution stand for 24 hours.

d) Neutralize and flush the solution down the drain with a large excess of water.

Summary of Method

Zinc and other metals in the sample complex with cyanide. Adding cyclohexanone selectively releases zinc. The zinc then reacts with the 2-carboxy-2'-hydroxy-5'-sulfoforamazyl benzene (zincon) indicator and forms a blue color that is proportional to the zinc concentration.

REQUIRED REAGENTS

Zinc Reagent Set, 20 mL size (100 tests) 24293-00
Includes: (1) 14033-32, (1) 21066-69

Description	Quantity Required Per Test	Units	Cat. No.
Cyclohexanone	0.5 mL	100 mL MDB	14033-32
ZincoVer 5 Reagent Powder Pillows	1 pillow	100/pkg	21066-69

REQUIRED APPARATUS

Pipet, serological, 10 mL	1	each	532-38
Pipet Filler, safety bulb	1	each	14651-00
Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
Squeezers, plastic dropper	1	20/pkg	21247-20

OPTIONAL REAGENTS

Bleach, household	1 gal	buy locally
Cylinder, graduated, mixing, 25mL	each	20886-40
Hydrochloric Acid Standard Solution, 6 N	500 mL	884-49
Nitric Acid, ACS	500 mL	152-49
Nitric Acid 1:1	500 mL	2540-49
Sodium Hydroxide Standard Solution, 5.0 N	50 mL SCDB	2450-26
Water, deionized	4 L	272-56
Zinc Standard Solution, 100 mg/L Zn	100 mL	2378-42
Zinc Standard Solution, PourRite ampule, 25 mg/L as Zn, 2mL	20/pkg	14246-20

OPTIONAL APPARATUS

Ampule Breaker, PourRite ampules	each	24846-00
Aspirator, vacuum	each	2131-00
Beaker, glass, 1000 mL	each	500-53
Cylinder, graduated, 100 mL	each	508-42
Cylinder, graduated, mixing, 250 mL	each	26362-46
Filter discs, glass, 47 mm	100/pkg	2530-00
Filter holder, 47 mm	each	2340-00
Flask, erlenmeyer, 250 mL	each	505-46
Flask, filtering, 500 mL	each	546-19

OPTIONAL APPARATUS (continued)

Description	Units	Cat. No.
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 1000 mL	each	14574-53
Hot plate, micro 115 V	each	12067-01
Hot plate, micro 230 V	each	12067-02
pH paper, 1 to 11 pH	5 rolls/pkg	391-33
pH meter, *Sens**ion**™1*, portable with electrode	each	51700-10
Pipet filler, safety bulb	each	14651-00
Pipet, serological, 2 mL	each	532-36
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet, TenSette, tips for 19700-01	50/pkg	21856-96
Pipet, TenSette, 1.0 to 10.0 mL	each	19700-10
Pipet, TenSette, tips for 19700-01	1000/pkg	21856-28
Pipet, TenSette, tips for 19700-10	50/pkg	21997-96
Pipet, TenSette, tips for 19700-10	250/pkg	21997-25
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 0.5 mL	each	14515-34

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.

By Phone:
6:30 a.m. to 5:00 p.m. MST
Monday through Friday
800-227-HACH (800-227-4224)

By Mail:
Hach Company
P. O. Box 389
Loveland, Colorado 80539-0389 U.S.A.

By FAX:
970-669-2932 (Hach Loveland)

Information Required:

- Hach account number (if available)
- Billing address
- Shipping address
- Your name and phone number
- Purchase order number
- Catalog number
- Brief description or model number
- Quantity

Technical and Customer Service

Hach Technical and Customer Service Department personnel are eager to answer questions about our products and their use and to take your orders. Specialists in analytical methods, they are happy to put their talents to work for you. Call **1-800-227-4224**.

International Customers

Hach maintains a network of dealers and distributors throughout the world.

In Canada
Hach Sales and Service Canada Ltd.
1313 Border Street, Unit 34
Winnipeg, Manitoba R3H 0X4
Telephone: (204) 632-5598
FAX: (204) 694-5134

In other countries, contact:
Hach Company World Headquarters
P. O. Box 389
Loveland, Colorado, U.S.A. 80539-0389
Telephone: (1) (970) 669-3050
FAX: (1) (970) 669-2932

Information presented on these pages applies only to Hach products manufactured for use within the United States. Exportation of these products renders these terms void.

Prices and Terms

Prices are subject to change without notice. All prices are FOB from the shipping point (usually Ames, Iowa). Hach offers instant credit up to $200 on Net 30 Day terms. Larger orders are subject to credit review. Customers may send remittance with orders or we can ship C.O.D. if you prefer.

Warranty

Hach warrants its products to be of high quality, to be free of material defects on the date of shipment and to be as specified.

Limits of Usage

Our chemicals and reagents are offered for laboratory and manufacturing use ONLY. They may not be used as drugs, cosmetics or food additives.

MSDS

Hach Material Safety Data Sheets, among the most complete and informative in the industry, provide comprehensive safety data essential for day-to-day operations and safety training.

An MSDS accompanies all Hach chemical products including test kits. For an additional cost, we will print MSDSs on your own forms.

ADDITIONAL INFORMATION

Label Information

Labels on Hach chemicals and reagents supply the following:

- Product Name -- In French, German, Italian and Spanish as well as English is printed on all but the smallest-size labels.
- Hach Catalog Number -- Makes reordering easy and helps match the appropriate MSDS.
- Storage Information and Lot Numbers -- Lot numbers made up of letters and numbers indicate an extended shelf life; a four-digit number indicates items should be rotated and checked with a standard to confirm performance. The lot number is essential if you call for technical assistance or with questions about reagent performance.

Shipping

Our experienced warehouse staff packages your orders for safe arrival. Unless we are instructed otherwise, the best and most efficient mode of transportation is selected. Motor freight shipments will be sent freight collect unless you specify otherwise at the time you order.

If you have questions about methods for shipment and availability of special packaging, please ask when you place your order.

Claims and Returns

We take extreme care to fill, check, re-check and pack orders properly. If errors or damages should occur, please report details to our Loveland Customer Service Department and to the carrier immediately. Be sure to keep all containers and packing materials.

AUTHORIZATION MUST BE OBTAINED from Hach when returning items for any reason. Call 1-800-227-4224 toll free. **ALL "FREIGHT COLLECT" SHIPMENTS OR MERCHANDISE RETURNED WITHOUT PROPER AUTHORIZATION FROM HACH WILL BE REFUSED.**

Multi Water Quality Checker
U-50 Series

Instruction Manual

CODE:GZ0000144342C

Preface

This manual describes the operation of the Multi Water Quality Checker, U-50 Series.
Be sure to read this manual before using the product to ensure proper and safe operation of the instrument. Also safely store the manual so it is readily available whenever necessary.

Product specifications and appearance, as well as the contents of this manual are subject to change without notice.

■ Warranty and Responsibility

HORIBA warrants that the Product shall be free from defects in material and workmanship and agrees to repair or replace free of charge, at HORIBA's option, any malfunctioned or damaged Product attributable to HORIBA's responsibility for a period of one (1) year from the delivery unless otherwise agreed with a written agreement. In any one of the following cases, none of the warranties set forth herein shall be extended;

- Any malfunction or damage attributable to improper operation
- Any malfunction attributable to repair or modification by any person not authorized by HORIBA
- Any malfunction or damage attributable to the use in an environment not specified in this manual
- Any malfunction or damage attributable to violation of the instructions in this manual or operations in the manner not specified in this manual
- Any malfunction or damage attributable to any cause or causes beyond the reasonable control of HORIBA such as natural disasters
- Any deterioration in appearance attributable to corrosion, rust, and so on
- Replacement of consumables

HORIBA SHALL NOT BE LIABLE FOR ANY DAMAGES RESULTING FROM ANY MALFUNCTIONS OF THE PRODUCT, ANY ERASURE OF DATA, OR ANY OTHER USES OF THE PRODUCT.

■ Trademarks

Generally, company names and brand names are either registered trademarks or trademarks of the respective companies.

Conformable Directive

This equipment conforms to the following directives and standards:

Directives: the EMC Directive 2004/108/EC

Standards: [the EMC Directive]
EN61326-1:2006 Class B, Portable test and measurement equipment

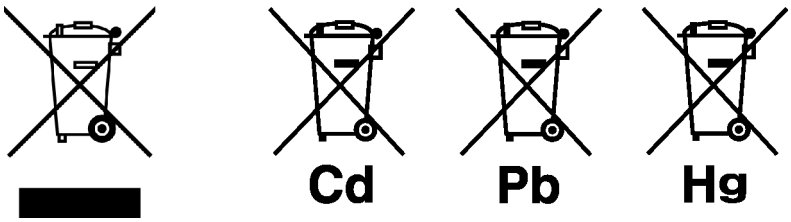
Information on Disposal of Electrical and Electronic Equipment and Disposal of Batteries and Accumulators

The crossed out wheeled bin symbol with underbar shown on the product or accompanying documents indicates the product requires appropriate treatment, collection and recycle for waste electrical and electronic equipment (WEEE) under the Directive 2002/96/EC, and/or waste batteries and accumulators under the Directive 2006/66/EC in the European Union.

The symbol might be put with one of the chemical symbols below. In this case, it satisfies the requirements of the Directive 2006/66/EC for the object chemical.

This product should not be disposed of as unsorted household waste.
Your correct disposal of WEEE, waste batteries and accumulators will contribute to reducing wasteful consumption of natural resources, and protecting human health and the environment from potential negative effects caused by hazardous substance in products.

Contact your supplier for information on applicable disposal methods.



FCC Rules

Any changes or modifications not expressly approved by the party responsible for compliance shall void the user's authority to operate the equipment.

WARNING

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications.
Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

For your safety

Warning messages are described in the following manner. Read the messages and follow the instructions carefully.

● Meaning of warning messages

⚠DANGER	This indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury. This signal word is to be limited to the most extreme situations.
⚠WARNING	This indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.
⚠CAUTION	This indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. It may also be used to alert against unsafe practices. Without safety alert indication of hazardous situation which, if not avoided, could result in property damage.

● Symbols

	Description of what should be done, or what should be followed
	Description of what should never be done, or what is prohibited

■ Safety Precautions

This section provides precautions to enable you to use the product safely and correctly and to prevent injury and damage. The terms of DANGER, WARNING, and CAUTION indicate the degree of imminency and hazardous situation. Read the precautions carefully as it contains important safety messages.

⚠ WARNING
Do not disassemble or modify the meter. May cause overheating or fire, resulting in accidents.

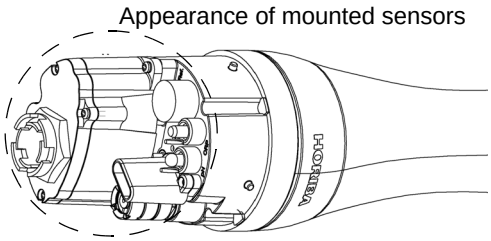
⚠ CAUTION
The pH and ORP sensors are made of glass. Handle them carefully to avoid breakage.
Do not ingest the DO, pH or ORP standard solutions. If it comes into contact with the eyes, rinse thoroughly with water. If swallowed, consult a physician.
Keep away from water when using USB communication. Improper use may result in fire or damage.

Points of concern

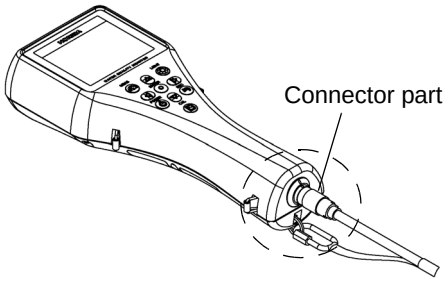
Use of the equipment in a manner not specified by the manufacturer may impair the protection provided by the equipment. It may also reduce equipment performance.

● Sensor probe

- Do not immerse the sensor probe in seawater or other samples with high salinity. Doing so may erode metallic parts. After use, promptly wash the sensor probe thoroughly in water.
- Do not immerse the sensor probe in alcohol, organic solvent, strong acid, strong alkaline, and other similar solutions.
- Do not subject to strong shocks.
- Do not perform measurement in environments of magnetic fields. Measurement errors may result.
- The sensor probe is no longer waterproof when the sensors are not mounted.

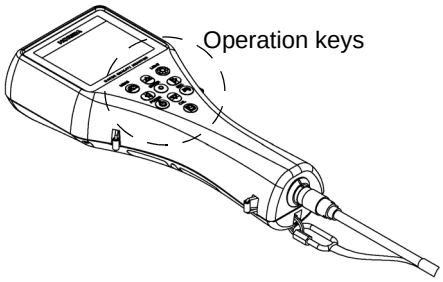


- Does not support measurement of samples containing fluorine.
- To disconnect the sensor cable or interface cable, pull them out with holding the connector part. Do not pull the cable part; it may cause breakage.



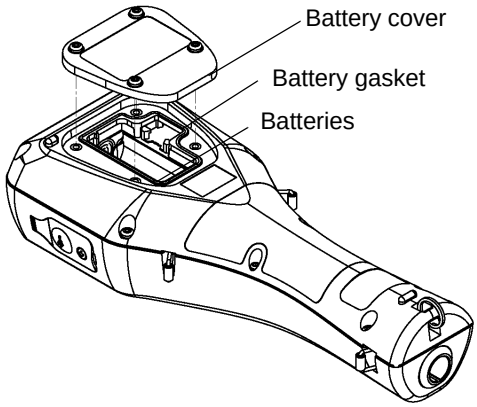
● Control unit

- Do not subject to strong shocks.
- The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.



- The control unit is no longer waterproof when the USB cable is connected.
- When operating the control unit only, protect the connector with the connector cap provided.

- Remove the batteries when not using the control unit for an extended period of time. Battery fluid leakage may cause equipment failure.
- Do not wipe the control unit with organic solvents or powder polish. The surface may deteriorate or its printing may disappear. If the display becomes dirty, wipe the dirt off with a soft cloth soaked in neutral detergent.
- Do not turn the power OFF or disconnect the cable during calibration or setting. Memory data may be erased.
- To perform measurement, connect the sensor probe cable before turning the power ON.
- Do not remove the battery gasket or twist it.
- When opening the battery case, make sure that no foreign matter is attached to the battery gasket.
- Do not use any unspecified batteries; it may cause breakage.



Measurement

- Do not pull the cable when lowering the sensor probe into the sample during measurement. Lower the sensor probe into the sample on a chain or string.
- Before lowering the sensor probe into the sample, do not connect the hook on the unit to a human body.
- The correct values are not displayed if the sensor is not mounted when the measurement display is activated.
- Perform DO measurement with no air bubbles in the internal solution.
- Do not reuse a membrane cap of DO sensor.
- Use the spanner for DO sensor provided to attach or remove the DO sensor.
- Avoid both U-53 and U-53G turbidity measurement in air, since the rubber wiper will quickly become damaged.
- Avoid turbidity measurement in direct sunlight, since the readout may be affected.

Calibration

During atmosphere calibration for the DO electrode with DO salinity compensation set to automatic, values are compensated based on electrical conductivity, but calibration is performed normally.

Location of use and storage

- Storage temperature: −10°C to 60°C
- Relative humidity: Under 80% and free from condensation

Store the meter in locations void of dust, strong vibrations, direct sunlight, corrosive gases, near air conditioners or windy areas.

Disposal of the product

When disposing of the product, follow the related laws and/or regulations of your country for disposal of the product.

Description in this manual

Note

This interprets the necessary points for correct operation and notifies the important points for handling the unit.

Reference

This indicates where to refer for information.

Tip

This indicates reference information.

Contents

1 About this Unit

The U-50 Series Multi Water Quality Checker features an integrated control unit and sensors. It is capable of making a maximum of eleven simultaneous measurements for various parameters, and is perfect for use in the field. The U-50 Series is designed with on-site ease-of-use in mind, provides a wide variety of functions, and can be used for water quality measurements and inspections of river water, groundwater, and waste water.

2 Device Information

2.1 Measurement parameters

Parameters	Model				
	U-51	U-52	U-52G	U-53	U-53G
pH (pH)	✓	✓	✓	✓	✓
pH (mV)	✓	✓	✓	✓	✓
Oxidation reduction potential (ORP)	✓	✓	✓	✓	✓
Dissolved oxygen (DO)	✓	✓	✓	✓	✓
Electrical conductivity (COND)	✓	✓	✓	✓	✓
Salinity (SAL) [expressed as electrical conductivity]	✓	✓	✓	✓	✓
Total dissolved solids (TDS) [expressed as electrical conductivity]	✓	✓	✓	✓	✓
Seawater specific gravity (SG) [expressed as electrical conductivity]	✓	✓	✓	✓	✓
Water temperature (TEMP)	✓	✓	✓	✓	✓
Turbidity (TURB) [LED transmission/front 30° scattering method]	–	✓	✓	–	–
Turbidity (TURB) [tungsten lamp 90° transmission/scattering method] with wiper	–	–	–	✓	✓
Water depth (DEP)	–	–	✓	✓	✓
GPS	–	–	✓	–	✓

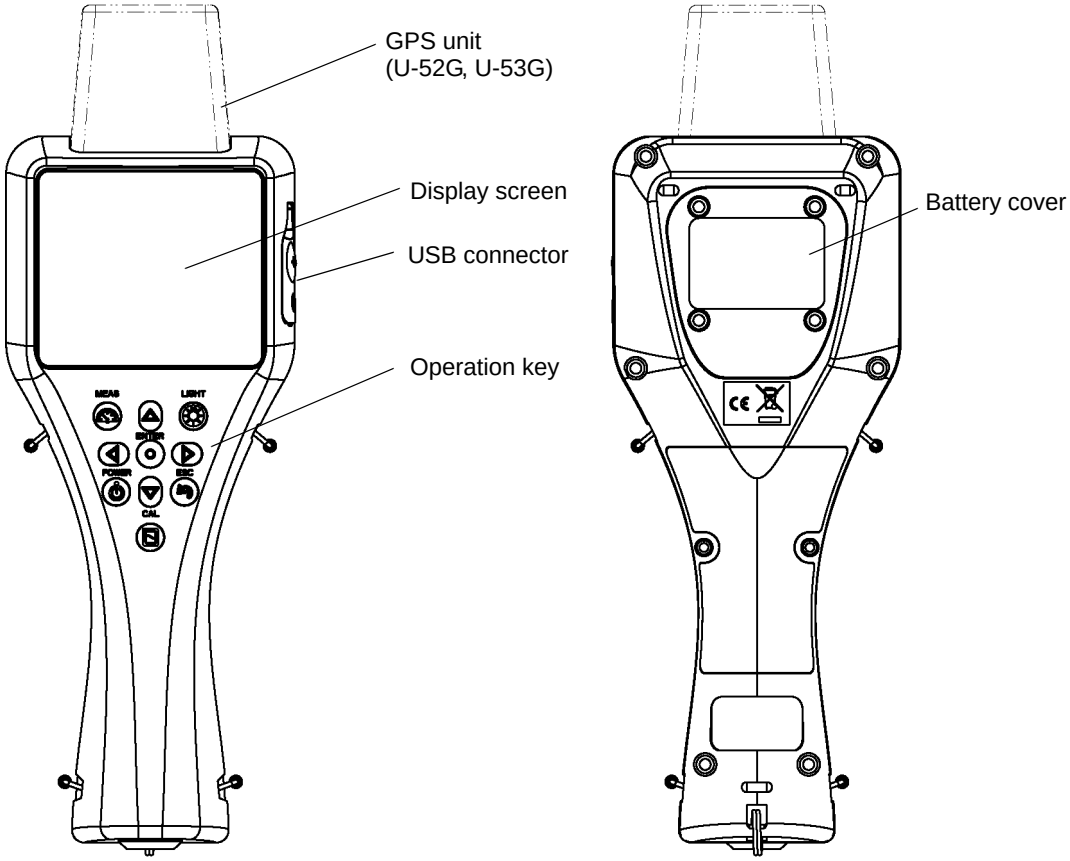
"✓" indicates a measurable parameter.

2.2 Packing list

Parts Name	Quantity	Note
Control unit	1	
Sensor probe	1	
pH sensor (#7112)	1	
ORP sensor (#7313)	1	
Reference electrode (#7210)	1	
DO sensor (#7543)	1	
Turbidity sensor (#7800)	1	With U-52/U-52G only. Attached to the sensor probe.
Turbidity sensor (#7801)	1	With U-53/U-53G only. Attached to the sensor probe.
pH 4 standard solution (#100-4)	1	500 mL
pH reference internal solution (#330)	1	250 mL
DO sensor internal solution set (#306)	1	Internal solution (50 mL), Sandpaper (#8000, #600), Syringe
DO Membrane spare parts set	1	
Spanner for DO sensor	1	
Cleaning brush	1	
calibration cup	1	transparent calibration cup, black calibration cup
Back pack	1	
Strap	1	
Alkaline batteries	4	LR14
Silicon grease	1	
Instruction manual	1	

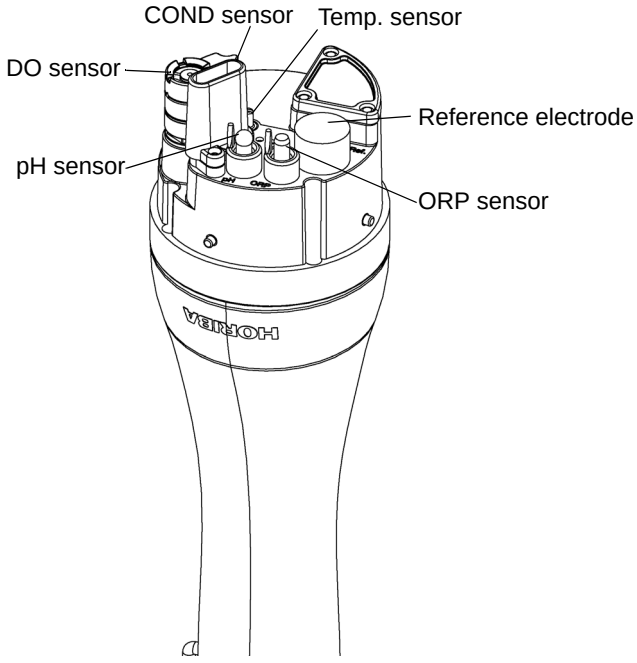
2.3 Parts name and functions

● Display

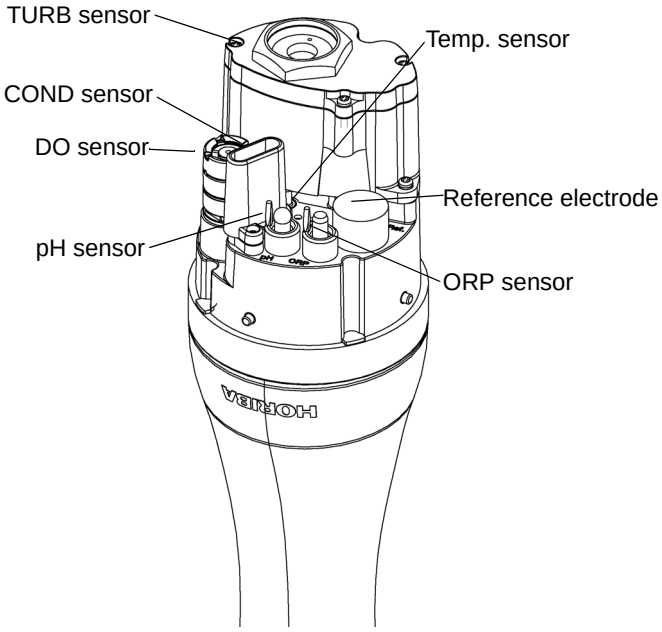


Front view　　Back view

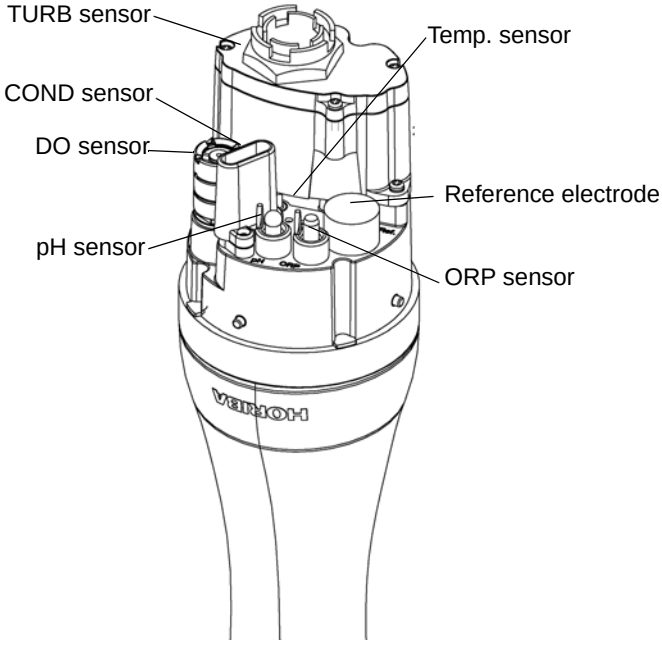
● Sensor probe (U-51)



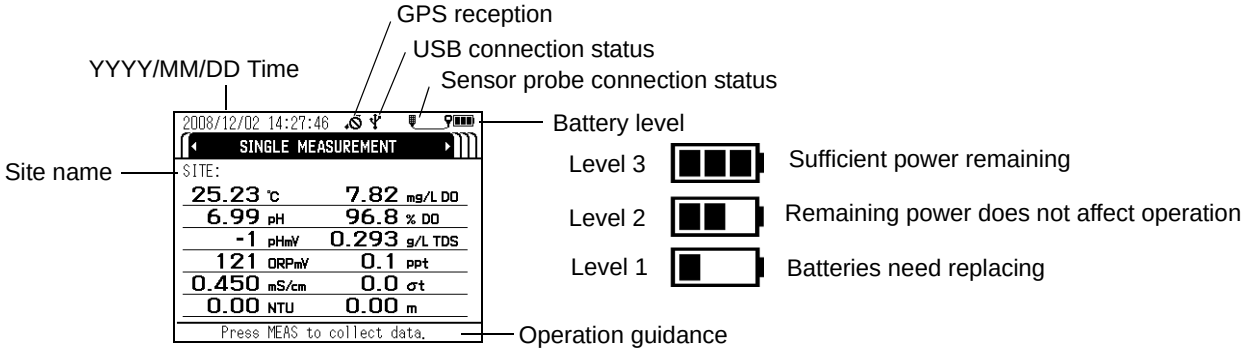
● Sensor probe (U-52)



● Sensor probe (U-53)



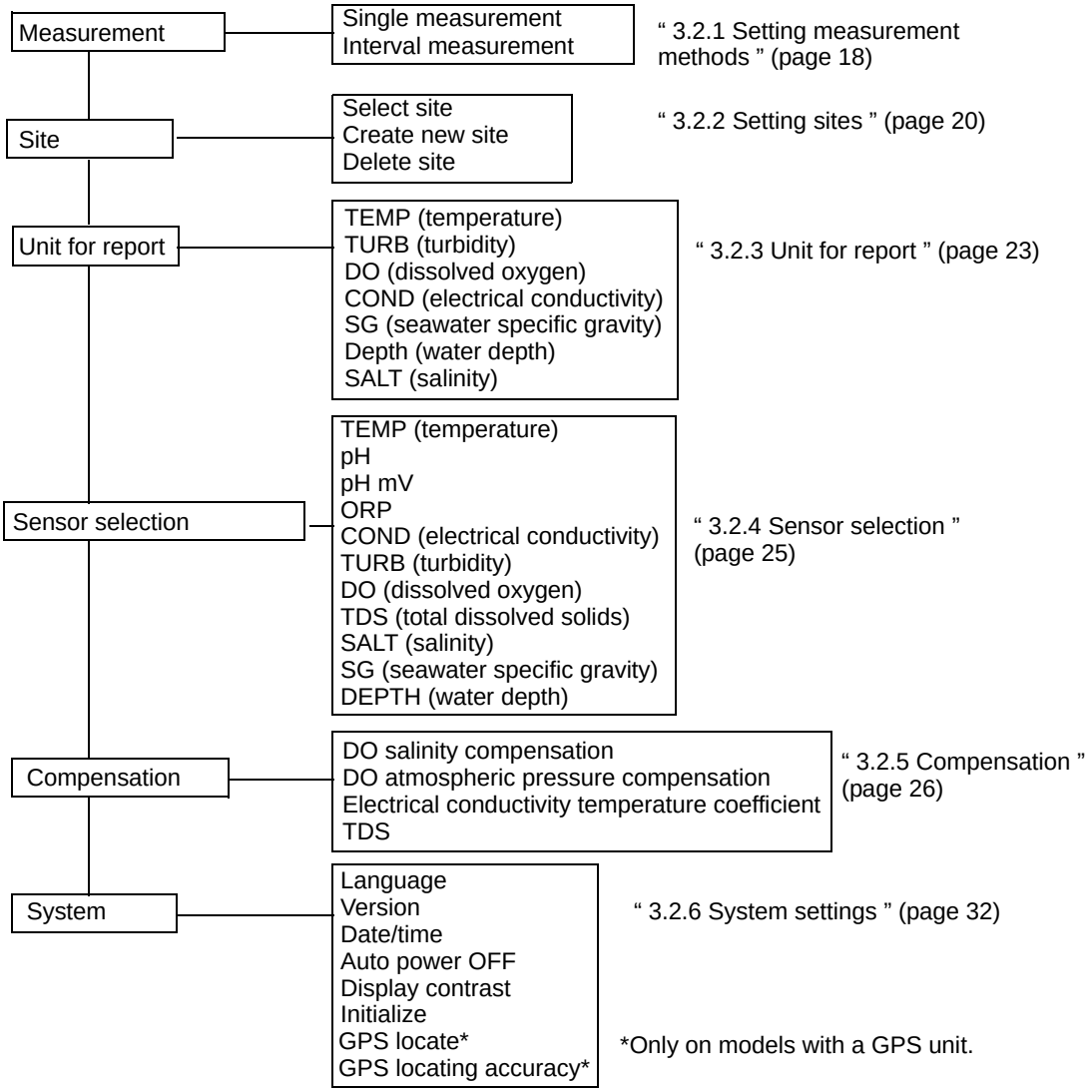
● Display screen



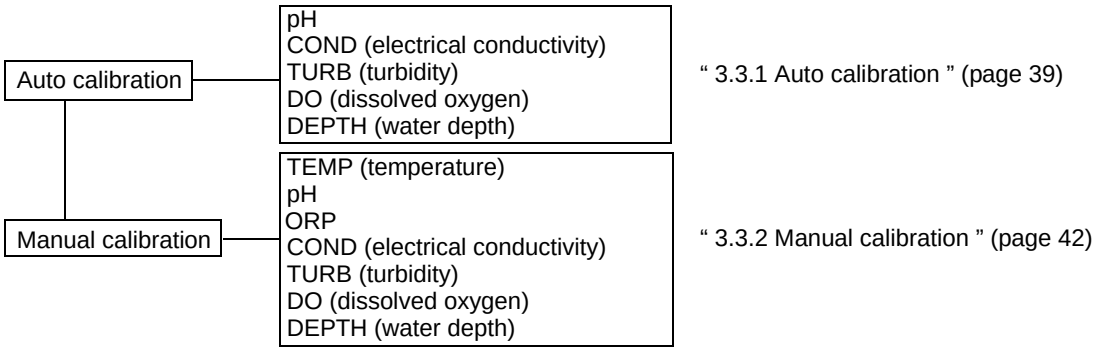
● Operation key

	Key name	description
	POWER key	Turns the system's power ON/OFF. The initial screen appears immediately after turning the power ON. Press and hold down the POWER key for about 3 seconds to turn the power ON and OFF.
	MEAS key	When pressed in the measurement screen, used to set the measurement values of all the measurement parameters. Measurement values flash until the data stabilizes.
		When pressed in the setting, calibration or data operation screen, returns to the measurement screen.
	ENTER key	Used to execute functions, set entered values or store data in memory.
	CAL key	Switches to the calibration screen.
	ESC key	Returns to the immediately preceding operation.
	LIGHT key	Turns the backlight ON/OFF. ● Using the backlight shortens battery life. ● The backlight does not light for about 3 seconds after power ON. ● When the sensor probe is connected while the display's backlight is lit, the backlight goes out for about 3 seconds.
	Left key	Moves the cursor to the left.
	Right key	Moves the cursor to the right.
	Up key	Moves the cursor up.
	Down key	Moves the cursor down.

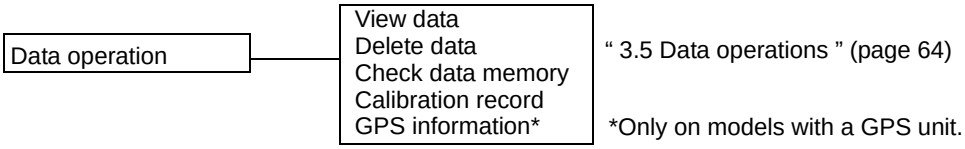
2.4 Setting menu items



2.5 Calibration menu items



2.6 Data operation menu items



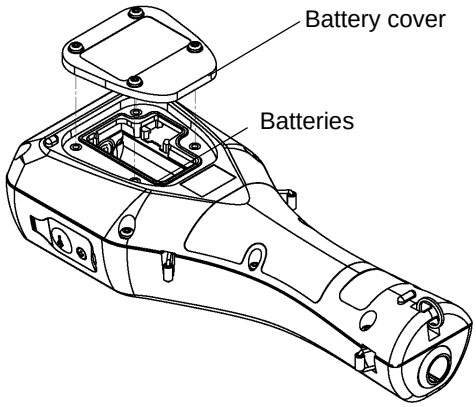
3 Basic Operation

3.1 System setup

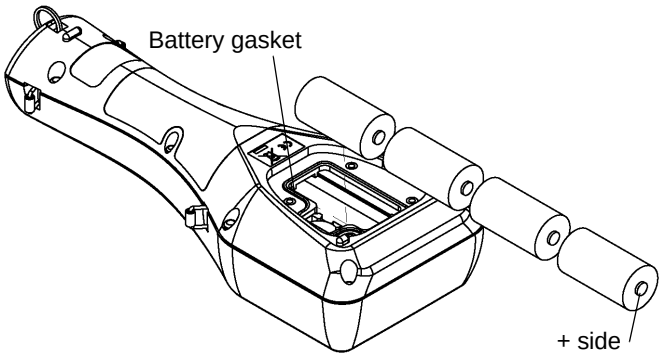
3.1.1 Inserting and replacing the batteries

The control unit is shipped without batteries. Follow the steps below to insert the batteries when using the system for the first time or replacing old batteries.

1. **Loosen the 4 screws on the battery cover by using No. 2 Phillips head screwdriver and remove the cover.**



2. **If replacing the batteries, discard the old batteries.**
3. **Insert new batteries in the control unit.**
   Check that the battery gasket is not dirty or twisted.



4. **Replace the battery cover and fasten it with the 4 screws.**
   Tighten the screws to less than 0.5 N·m.

Note

- **Data and settings will not be lost when the batteries are replaced.**
- **If dirty or twisted, the battery gasket will fail to keep the batteries dry. Check its condition before closing the cover.**
- **To ensure long service life, replacing the battery gasket periodically (once a year) is recommended.**

Precautions when using dry cell batteries

- **Batteries to use: LR14 alkaline dry cell batteries (C-size dry cell batteries) or rechargeable nickel-metal hydride dry cell batteries (C-size)**
  Do not use manganese batteries.
- **Dry cell batteries used incorrectly may leak or burst. Always observe the following**
  - Orient the batteries correctly (positive and negative ends in correct positions).
  - Do not combine new and used batteries, or batteries of different types.
  - Remove the batteries when not using the system for a prolonged period.
  - If batteries leak, have the system inspected at your nearest Horiba service station.

● Battery life

- The battery life for continuous operation when using C-size alkaline dry cell batteries is about 70 hours.
- Using the backlight consumes a proportionate amount of battery power, shortening battery life.
- Searching position information using the GPS unit consumes a proportionate amount of battery power, shortening battery life.
- Nickel-metal hydride secondary batteries can be used, but the battery life is not guaranteed since it will vary according to usage (number of times data is saved, number of charges and amount of each charge). In general, secondary batteries have one-half to one-third the life of C-size alkaline batteries.
- The 70-hour battery life figure applies to a control unit operating temperature of 20°C or more. The battery characteristics shorten the battery life at operating temperatures lower than 20°C, so check the remaining battery level, and replace the batteries before it reaches Level 1.
- The batteries packed with the system at the time of shipment are for checking operation. Their life is not guaranteed.
- The 70-hour battery life figure is the amount of operating time the batteries can provide until the system stops operating. The system may fail during operation if the remaining battery level is low, so it is a good idea to check the remaining battery level and replace the batteries with new ones well before the batteries run out completely.

U-51/52

Battery life: 70 hours (backlight off)

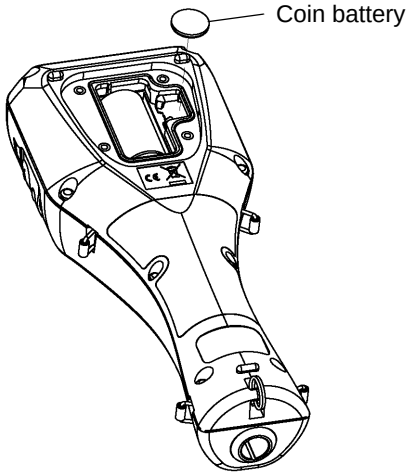
U-53

Battery life: 500 measurements (backlight off)

- Since U-53 is designed for turbidity measurement with wiper, its battery life is estimated in terms of the number of turbidity measurement sequences performed.
- Battery power is also consumed by measurement operations other than turbidity measurement.
- The battery life when turbidity measurement is not performed is about 70 hours.

3.1.2 Replacing the coin battery

- Coin battery to use: CR-2032
- The coin battery is only for the clock. It will provide problem-free operation for three years, but when using the clock continuously, it should be replaced every two years as a precaution.
- When replacing the coin battery for the clock, leave the control unit ON. If the coin battery is replaced when the control unit is turned OFF, the clock will be reset to the default settings.



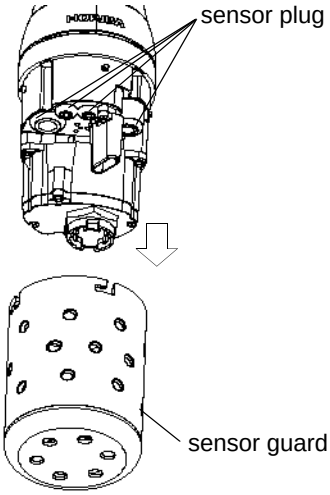
3.1.3 Attaching sensors

Note

- **When attaching or replacing a sensor, wipe any moisture off the sensor probe and sensor.**
- **Be sure to keep water out of sensor connectors. If moisture comes in contact with a sensor connector, blow-dry it with dry air.**
- **The sensor probe is not waterproof when the sensor is not mounted.**
- **Take care not to tighten the sensor too much.**

● Attaching the pH sensor

1. **Remove the sensor guard.**



2. **Remove the sensor plug.**

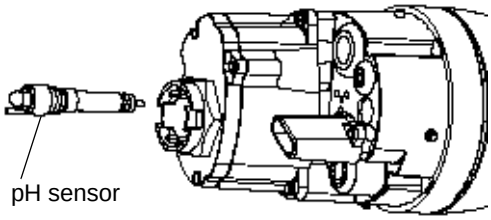
3. **Coat the pH sensor O-ring with a thin layer of silicon grease (part No. 3014017718).**

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

4. **Make sure there is no moisture on the sensor probe's sensor connector (marked "pH").**

5. **Fasten the pH sensor securely by hand.**



6. **Clean the sensor with an alcohol-soaked cloth.**

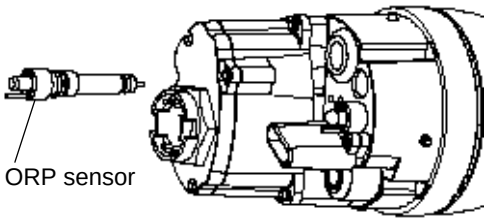
Attaching the ORP sensor

1. **Remove the sensor guard.**
2. **Remove the sensor plug.**
3. **Coat the ORP sensor O-ring with a thin layer of grease (part No. 3014017718).**

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

4. **Make sure there is no moisture on the sensor probe's sensor connector (marked "ORP").**
5. **Fasten the ORP sensor securely by hand.**



6. **Clean the sensor with an alcohol-soaked cloth.**

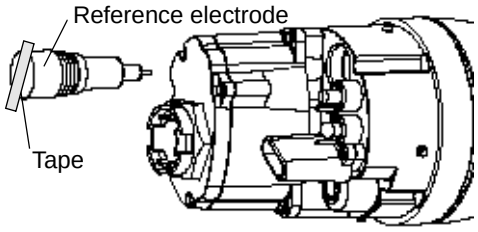
Attaching the reference electrode

1. **Remove the sensor guard.**
2. **Remove the sensor plug.**
3. **Coat the reference electrode O-ring with a thin layer of grease (part No. 3014017718).**

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

4. **Make sure there is no moisture on the sensor probe's sensor connector (marked "REF").**
5. **Fasten the reference electrode securely by hand.**
6. **Remove the tape from the liquid junction part of the reference electrode.**



Attaching the dissolved oxygen (DO) sensor

1. **Remove the membrane cap mounted on the DO sensor beforehand, and replace it with the new membrane cap provided. Replace the internal solution with fresh solution. The main component of the internal solution is potassium chloride (KCl), so the old solution can be disposed of down a sink or other drain.**

Reference

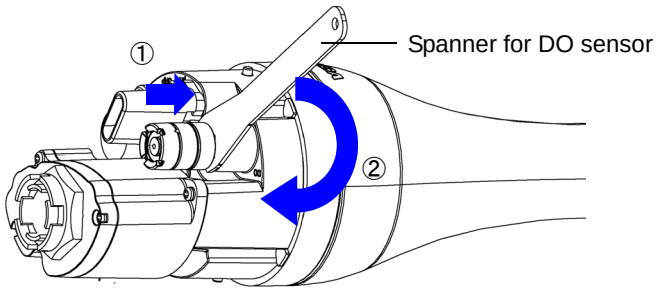
“ 4.5 Replacing the membrane cap ” (page 87)

2. **Screw in the DO sensor to attach it, allowing the internal solution to overflow slightly.**
3. **Use a soft cloth to wipe off the internal solution that overflowed onto the DO sensor.**
4. **Remove the sensor guard.**
5. **Remove the sensor plug.**
6. **Coat the DO sensor O-ring with a thin layer of grease (part No. 3014017718).**

Note

Be sure no grease from the O-ring gets on the sensor connector. If the sensor connector gets grease on it, wipe it off with a soft cloth soaked in alcohol.

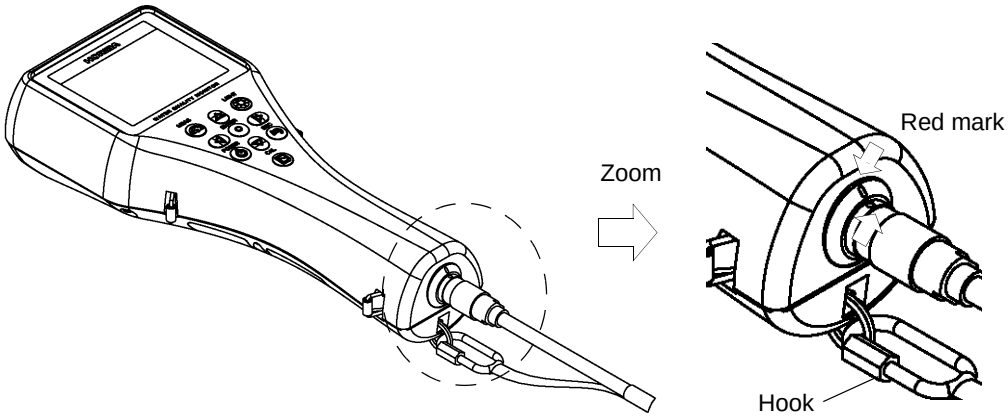
7. **Make sure there is no moisture on the sensor probe’s sensor connector (marked "DO").**
8. **Fasten the DO sensor securely using the spanner for DO sensor.**
   - Hold the DO sensor with the provided spanner for DO sensor and push the sensor down. (Step 1 in figure below)
   - Screw the DO sensor in place. (Step 2 in figure below)



3.1.4 Connecting the control unit and sensor probe

Note

Connect the control unit with its power OFF.

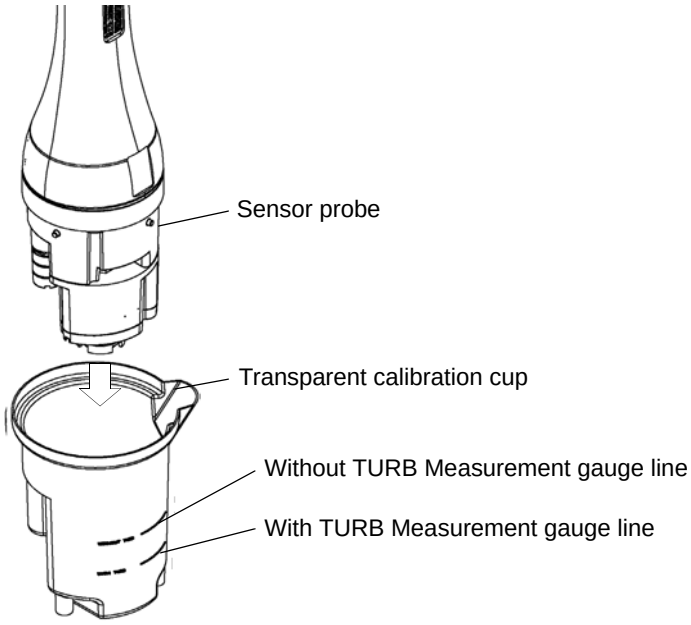


1. **Align the red mark on the connector, and press the connector in until you hear it click.**
2. **Connect the cable's hook to the display.**

3.1.5 Conditioning

Carry out the steps below when using the unit for the first time or when the system has not been used for 3 months or longer.

1. **Fill the transparent calibration cup to the line with pH 4 standard solution.**
   The transparent calibration cup has With TURB Measurement and Without TURB Measurement gauge lines.
2. **Insert the sensor probe in the transparent calibration cup.**



Note

Check that all sensors are attached.

3. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON. Leave the unit for at least 20 minutes to condition the sensors.**

Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

Tip

- **The procedure for immersing the sensor probe in the pH standard solution is the same as that described in " 3.3.1 Auto calibration " (page 39).**
  Auto calibration can be performed using the same pH 4 standard solution that was used in the conditioning procedure.
- **Immersing the sensor in the standard solution is generally required for sensor conditioning, but a voltage supply is required for DO sensor conditioning. Turning ON the power of the control unit is necessary during sensor conditioning.**

3.1.6 GPS (U-52G, U-53G)

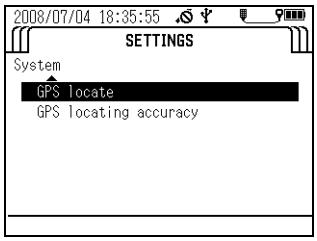
The GPS position measurement precision is proportional to the GPS position measurement time. When the position measurement precision increases, the position measurement time also increases. See " ● GPS locating accuracy" (page 17) for how to set the position measurement precision. See " ● GPS locate" (page 15) below for how to check acquired GPS data.

● GPS locate

1. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

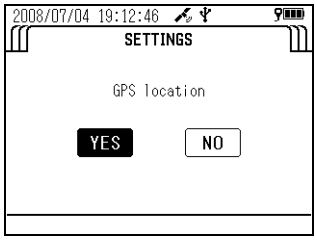
2. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**

3. **Press the down (▽) key to move the cursor to "GPS locate", then press the ENTER key.**



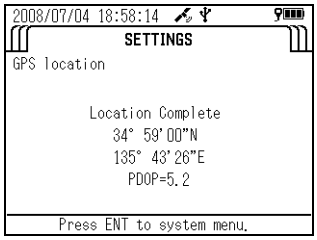
4. **The message "Press ENT key to start position measurement." appears. Press the ENTER key.**

5. **The message "Execute GPS position measurement?" appears. Move the cursor to "YES", then press the ENTER key.**

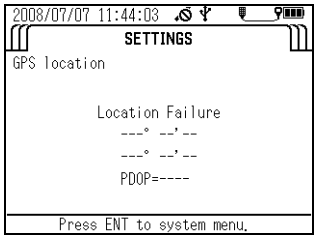


6. **The message "Warming up. Please wait." appears. Wait until the system has finished warming up (about 10 seconds).**

- Position measurement starts automatically when warmup has finished. Position measurement is performed up to 10 times.
- The GPS location complete screen appears after successful position measurement.

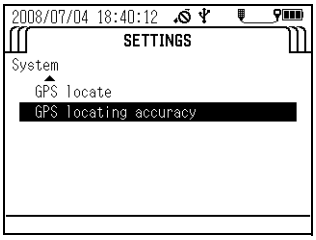


- The GPS location failure screen appears after position measurement has failed. Redo the measurement in a location free from obstacles, or wait for the meteorological conditions to improve before redoing the measurement.

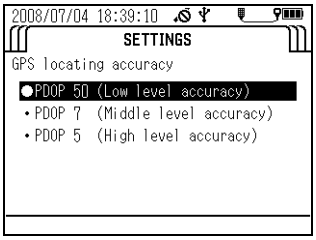


● GPS locating accuracy

1. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
2. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**
3. **Press the down (▽) key to move the cursor to "GPS locating accuracy", then press the ENTER key.**



4. **The screen below appears. Move the cursor to the locating accuracy, then press the ENTER key. The black circle (●) indicates the currently set precision.**



3.2 Settings

3.2.1 Setting measurement methods

This section describes how to set the measurement method.

● Measurement methods

● U-51/U-52

Single measurement	Pressing the MEAS key acquires the 5-second average for the selected measurement parameter.
Interval measurement	Pressing the MEAS key acquires and saves the 5-second average for the selected measurement parameter in the set interval. The measurement interval can be set to any value between 10 seconds and 24 hours.

● U-53

The U-53 turbidity sensor uses a tungsten lamp. The lamp lights for about 10 seconds, and the average measurement value acquired during this interval is displayed.

Single measurement	Pressing the MEAS key acquires the 5-second average for the selected measurement parameter after wiper operation. The 10-second average is acquired when measuring turbidity.
Interval measurement	Pressing the MEAS key acquires and saves the 5-second average for the selected measurement parameter in the set interval. The 10-second average is acquired when measuring turbidity. The measurement interval can be set to any value between 10 seconds (final check of this value required; 30 seconds may be better for U-52) and 24 hour.

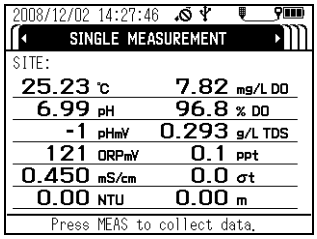
Reference

“ 3.4 Measurement ” (page 61)

● Operation method

1. **Press and hold down the control unit’s POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

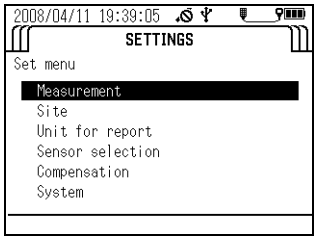


Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

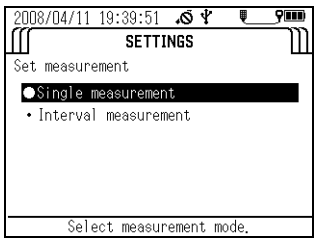
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "Measurement", then press the ENTER key.**



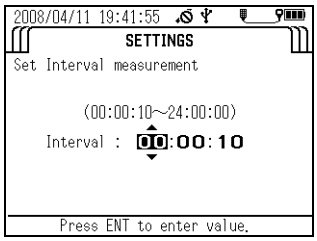
4. **Press the down (▽) key to move the cursor to the desired measurement mode. Press the ENTER key to save the setting.**

The black circle (●) indicates the currently selected measurement mode.



5. **If you selected "Interval measurement", the display switches to the screen used to set the measurement interval. Press the up (△) and down (▽) keys to set the measurement interval.**

The measurement interval can be set to any value between 10 seconds and 24 hours in the case of the U-51 and U-52, or between 30 seconds and 24 hours in the case of the U-53.



3.2.2 Setting sites

The site function allows position data to be connected to corresponding measurement data. Sites have the following specifications and features:

Site names: Text data consisting of up to 20 one-byte alphanumeric characters, spaces, etc.
Site names can be used for control unit searches and as labels for computer processing.
Site names allow measurement data to be saved with a name corresponding to the actual location where it was measured.

You can use site information as a search key when viewing data uploaded by a PC or data saved in the control unit (see " 3.5 Data operations " (page 64)).

- **Selecting sites**

  You can select previously created sites. The black circle (●) indicates the name of the currently selected site. No sites are created at new purchasing or after initialization. Select a site after first creating one from the "Create new site" menu.

- **Creating new sites**

  You can create and save new sites. Up to 20 site names can be registered.

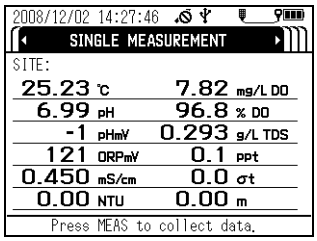
- **Deleting sites**

  You can select a previously created site and delete it.

● **Operation methods**

- **Selecting a site**

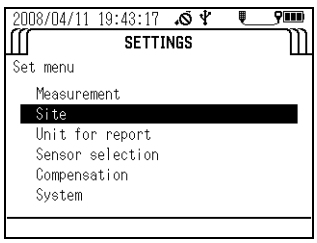
  1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
     The "MEASUREMENT" screen appears after about 10 seconds.



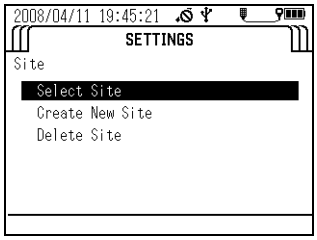
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

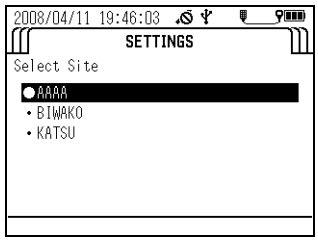
  2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
  3. **Press the down (▽) key to move the cursor to "Site", then press the ENTER key.**



4. **Press the down (▽) key to move the cursor to "Select Site", then press the ENTER key to display the names of the currently saved sites.**



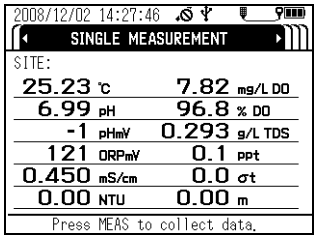
The black circle (●) indicates the currently selected site.



● **Creating a new site**

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

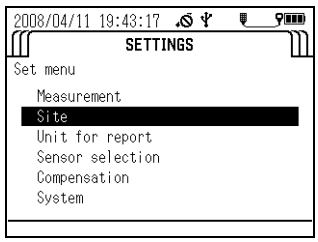


Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

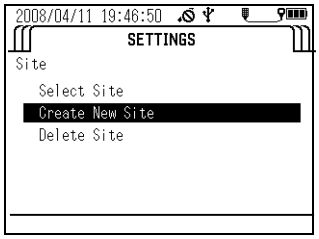
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "Site", then press the ENTER key.**

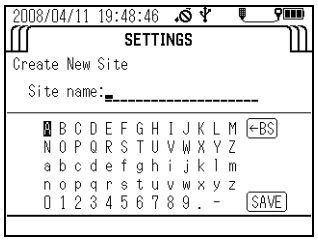


4. **Press the down (▽) key to move the cursor to "Create New Site", then press the ENTER key.**

Enter the desired site name (up to 20 alphanumeric non-Asian width characters).



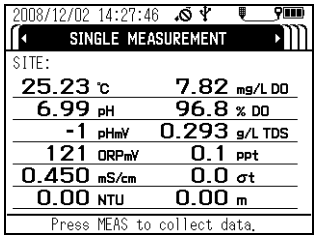
5. **Press the up (△), down (▽), right (▷), and left (◁) keys to move the cursor to each letter or number to use in the name, then press the ENTER key to confirm the entered characters. To delete incorrectly entered characters, move the cursor to "BS" and press the ENTER key to start deleting from the last character. When you have finished entering the name, save it by moving the cursor to "SAVE" and pressing the ENTER key.**



● **Deleting a site**

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

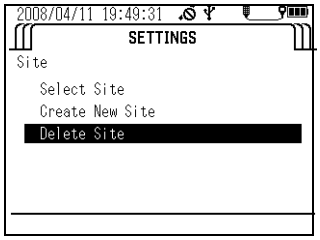
The "MEASUREMENT" screen appears after about 10 seconds.



Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

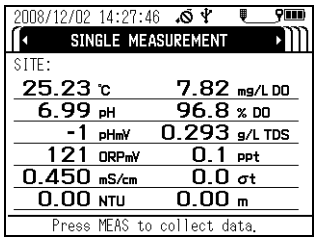
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**



3. **Press the down (▽) key to move the cursor to "Site", then press the ENTER key.**

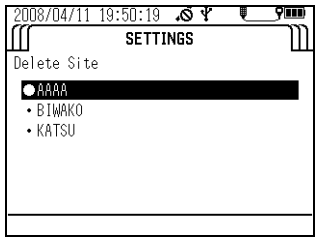
4. **Press the down (▽) key to move the cursor to "Delete Site", then press the ENTER key.**

A list of the currently saved sites appears. The black circle (●) indicates the currently selected site.



5. **Press the down (▽) key to move the cursor to the site to delete, then press the ENTER key to delete it.**

The currently selected site can be deleted after a different site has been selected from the site selection menu or after all unselected sites have been deleted. The same site name cannot be registered more than once.



3.2.3 Unit for report

Note

Units can only be selected when the sensor probe is connected.

Follow the steps below to set the measurement units of measurement parameters. No units are displayed if a measurement parameter has not been selected in the measurement parameter selection screen (see “ 3.2.4 Sensor selection ” (page 25)).

1. **Press and hold down the control unit’s POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

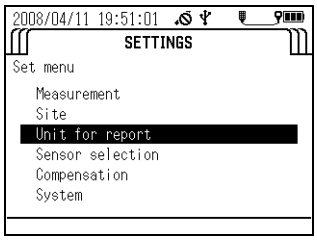
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

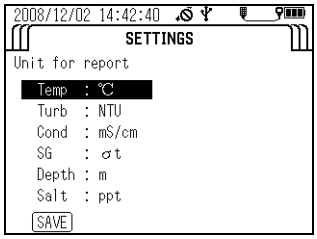
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "Unit for report", then press the ENTER key.**

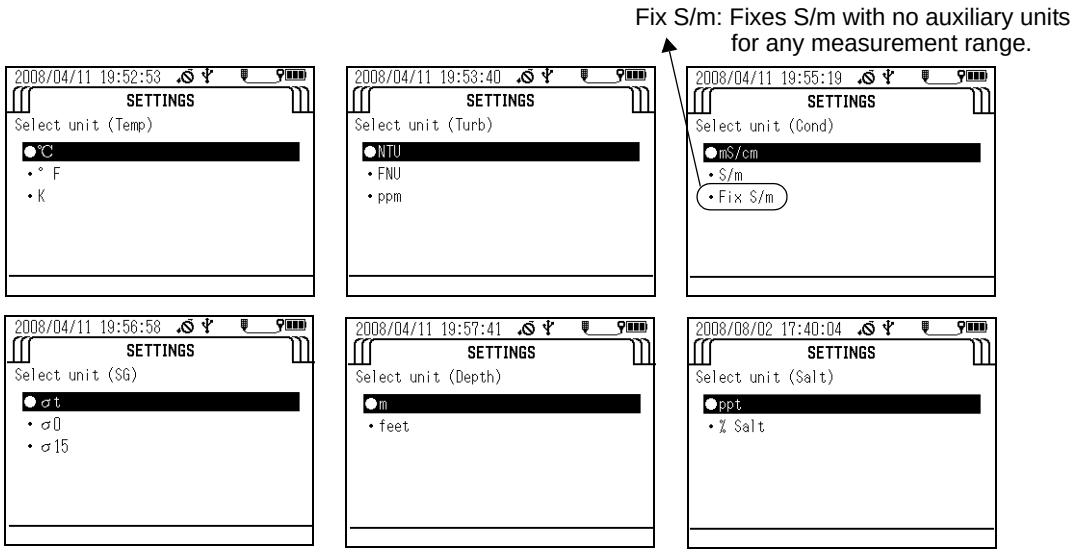
A list of the currently selected measurement parameters and their units appears. Note that measurement parameters not selected (in the measurement parameter selection screen) are not displayed.



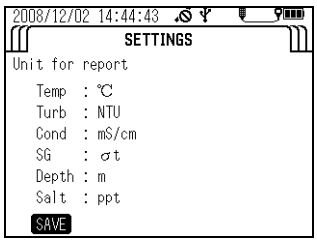
4. **Press the up (△) and down (▽) keys to move the cursor to the item to change, then press the ENTER key.**



5. **A list of the units that can be selected appears. The black circle (●) indicates the currently selected unit. Press the up (△) and down (▽) keys to move the cursor to the desired unit, then press the ENTER key.**



6. **To save the changes, press the up (△) and down (▽) keys to move the cursor to SAVE, then press the ENTER key. If you do not want to save the changes, press the ESC key.**



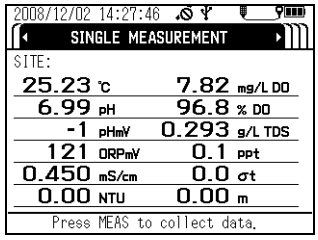
3.2.4 Sensor selection

Note

Measurement parameters can only be selected when the sensor probe is connected.

You can set between 1 and 11 measurement parameters to display in the control unit screen. Follow the steps below to select the desired measurement parameters.

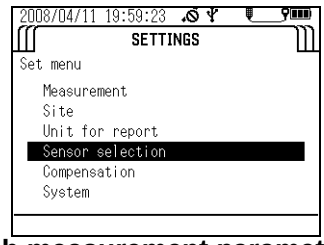
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.



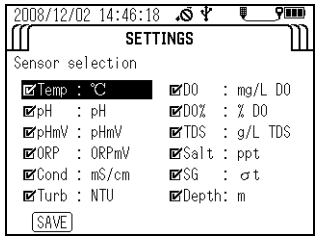
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "Sensor selection", then press the ENTER key.**
   A list of the measurement parameters that can be set and the currently set units are displayed.



4. **Move the cursor to each measurement parameter to change, then press the ENTER key.**
   A check in the check box of a measurement parameter indicates it will be displayed.
5. **To save the changes, press the up (△), down (▽), left (◁) and right (▷) keys to move the cursor to SAVE, then press the ENTER key. If you don't want to save the changes, press the ESC key.**



Note

Available measurement parameters differ according to product specifications.

3.2.5 Compensation

Note

Compensation settings can only be made when the sensor probe is connected.

U-50 series have following functions of compensation.

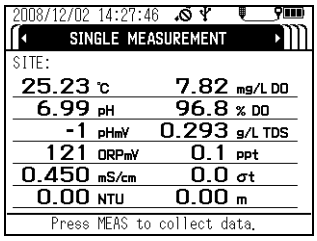
- Salinity compensation and atmospheric pressure compensation for dissolved oxygen (DO)
- Temperature compensation for conductivity (COND)
- Setting total dissolved solid (TDS) coefficient for TDS

● Salinity compensation (DO)

The dissolved oxygen (DO) value is presented higher than actual value if salinity compensation is not added, because the increase of salinity gives higher DO value. To obtain correct value salinity compensation is needed. The following modes are available for calculation of salinity compensation.

AUTO:	Salinity compensation is performed automatically with salinity converted from conductivity.
Value input:	Press the up (△) and down (▽) keys to enter a setting value when the salinity is known.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

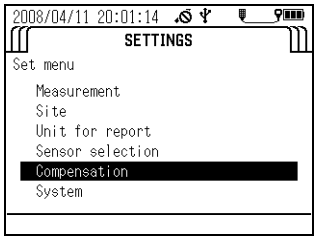


Note

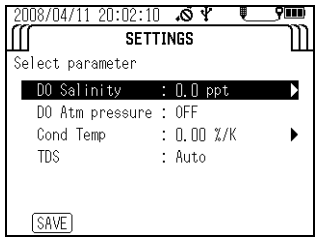
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

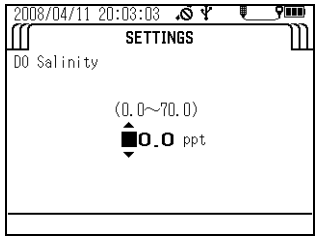
3. **Press the down (▽) key to move the cursor to "Compensation", then press the ENTER key.**



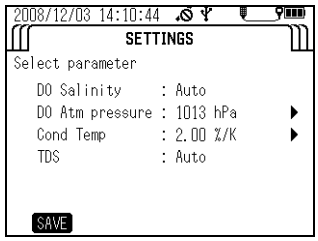
4. **Press the down (▽) key to move the cursor to "DO Salinity", then press the ENTER key to toggle the setting between "Auto" and "Input mode".**
Default: Auto



5. **If you selected "Input mode", press the right (▷) key to display the compensation value input screen. Press the up (△) and down (▽) keys to enter the desired value, then press the ENTER key to set it.**



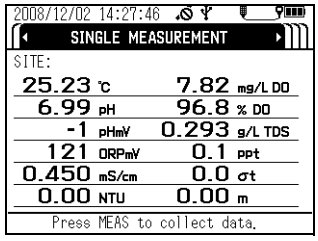
6. **To save the change, press the up (△) and down (▽) keys to move the cursor to SAVE, then press the ENTER key. If you don't want to save the change, press the ESC key.**



● **Atmospheric pressure compensation (DO)**

Differences in the atmospheric pressure of the measurement location influence the Dissolved Oxygen (DO) measurement. By setting (input) the actual atmospheric pressure of the measurement location into the control unit, it is possible to standardize the measured Dissolved Oxygen (DO) value to a value at the standard atmospheric pressure (1013 hPa).

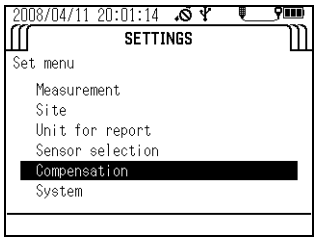
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.



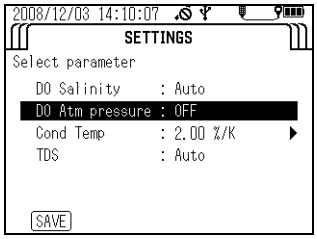
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

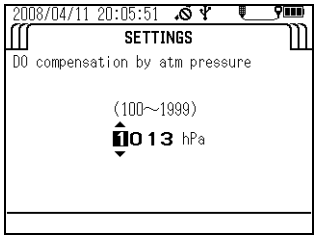
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "Compensation", then press the ENTER key.**



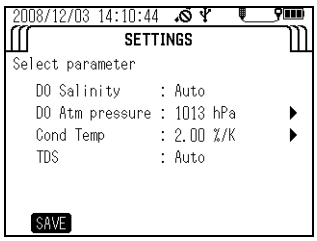
4. **Press the down (▽) key to move the cursor to "Cond Temp", then press the ENTER key to toggle the setting between "OFF" and "Input mode".**
   Default: OFF



5. **If you selected "Input mode", press the right (▷) key to display the compensation value input screen. Press the up (△) and down (▽) keys to enter the desired value, then press the ENTER key to set it.**



6. **To save the change, press the up (△) and down (▽) keys to move the cursor to SAVE, then press the ENTER key. If you don't want to save the change, press the ESC key.**

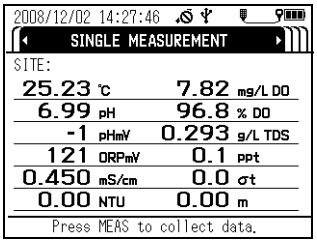


● Temperature compensation for conductivity (COND)

Sample conductivity (COND) varies with temperature, and this control unit uses a temperature compensation coefficient to automatically standardize the conductivity (COND) at 25°C. The initial setting coefficient is 2%/K, which is the generally used.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

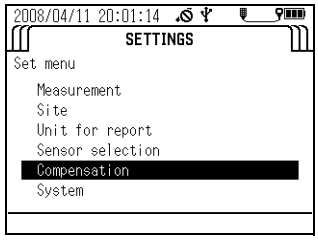


Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

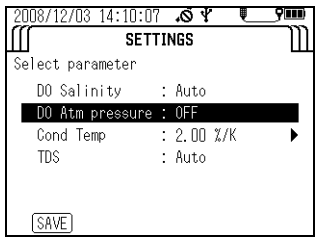
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "Compensation", then press the ENTER key.**

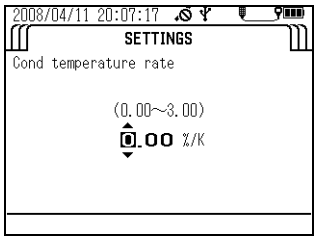


4. **Press the down (▽) key to move the cursor to "Cond Temp", then press the ENTER key to toggle the setting between "OFF" and "Input mode".**

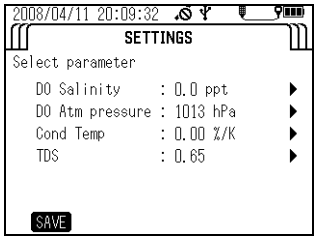
Default: 2.00%/K



5. **If you selected "Input mode", press the right (▷) key to display the compensation value input screen. Press the up (△) and down (▽) keys to enter the desired value, then press the ENTER key to set it.**



6. **To save the change, press the up (△) and down (▽) keys to move the cursor to SAVE, then press the ENTER key.**
If you don't want to save the change, press the ESC key.



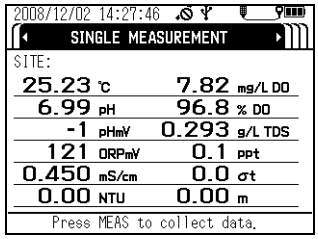
● Setting a total dissolved solid (TDS) coefficient

The total dissolved solid amount (TDS) is a converted value obtained by multiplying the conductivity (COND) by a known coefficient. The coefficient initially set for the control unit is based on a conversion for KCl and $CaCO_3$ solutions and it depends on the conductivity (COND) value as shown below.

Conductivity (COND) (S/m)	Conversion coefficient
< 0.05	0.65
0.05 to 0.5	0.64
0.5 to 1	0.63
1 to 3	0.62
3 to 5	0.61
> 5	0.60

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

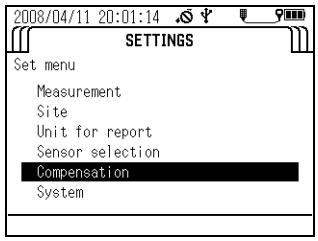


Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

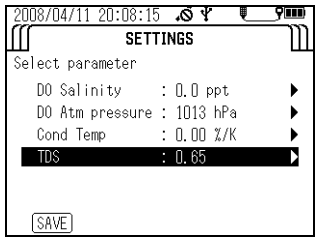
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "Compensation", then press the ENTER key.**

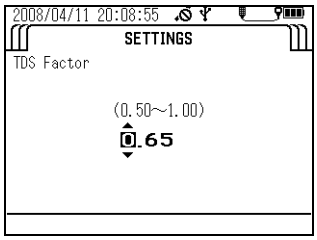


4. **Press the down (▽) key to move the cursor to "TDS", then press the ENTER key to toggle the setting between "AUTO" and "Input mode".**

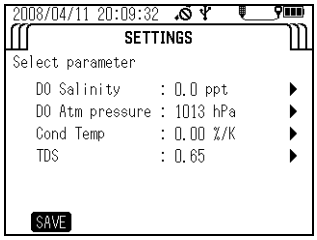
Default: Auto



5. **If you selected "Input mode", press the right (▷) key to display the compensation value input screen. Press the up (△) and down (▽) keys to enter the desired value, then press the ENTER key to set it.**



6. **To save the change, press the up (△) and down (▽) keys to move the cursor to SAVE, then press the ENTER key. If you don't want to save the change, press the ESC key.**



3.2.6 System settings

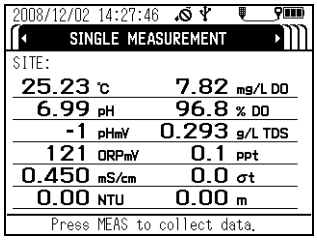
The system settings let you change the display language, check the system software version, set the date/time, set the auto power OFF time, set the display contrast, and initialize the settings.

● **Display language**

Follow the steps below to select either English or Japanese as the display language.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

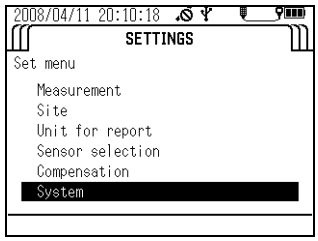


Note

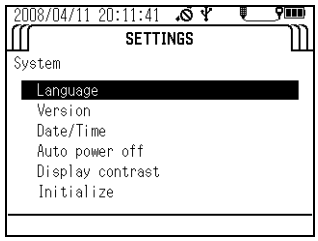
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**

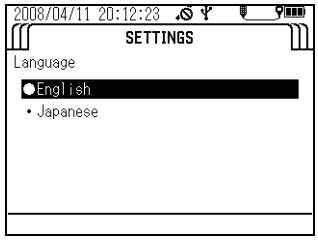


4. **Press the down (▽) key to move the cursor to "Language", then press the ENTER key.**



5. **A list of the supported display languages appears. Press the up (△) and down (▽) keys to move the cursor to the desired language, then press the ENTER key.**

The black circle (●) indicates the currently selected display language.



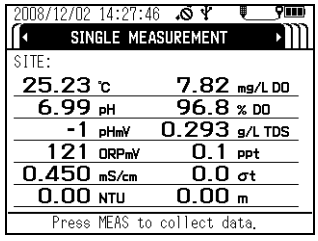
● Version

Follow the steps below to display the program No. and version of the control unit and sensor probe software.

The program No. and version of the sensor probe software will not be displayed if the sensor probe is not connected.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

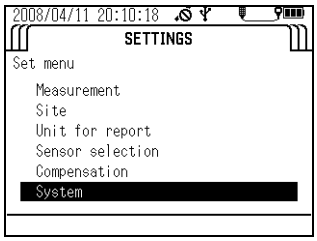


Note

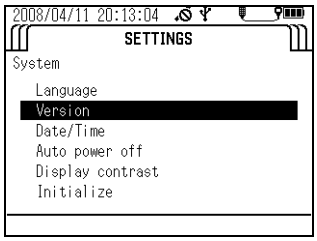
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**



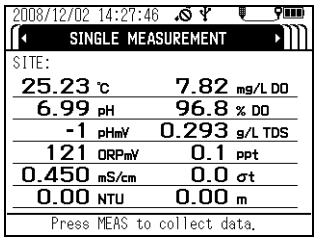
4. **Press the down (▽) key to move the cursor to "Version", then press the ENTER key.**
The program No. of the control unit and sensor probe software appears.



● Setting the date/time

Follow the steps below to set the date and time.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

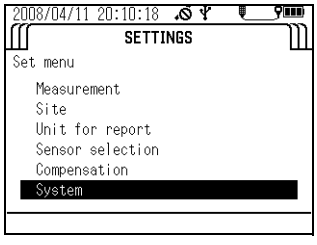


Note

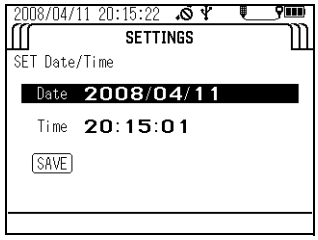
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**

3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**

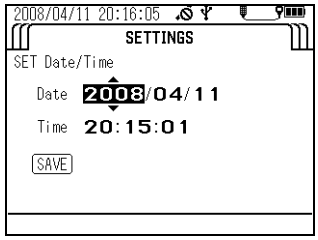


4. **Press the down (▽) key to move the cursor to "Date/time", then press the ENTER key.**

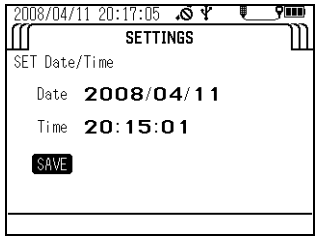


5. **Move the cursor to the date, then press the ENTER key.**

6. **Press the right (▷) key to move the cursor to the year, month, day, hour, minute and second, and press the up (△) and down (▽) keys to enter each value.**



7. **When finished entering settings, press the ENTER key to move the cursor to SAVE, then press the ENTER key again to save the settings.**

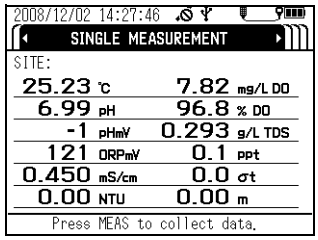


● Setting the auto power OFF time

Follow the steps below to set the time for the auto power OFF function (which turns the power OFF automatically when no operation is performed for the preset amount of time).

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

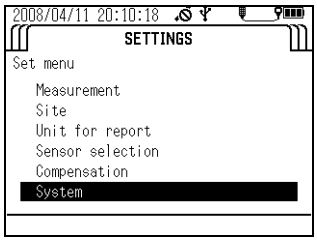
The "MEASUREMENT" screen appears after about 10 seconds.



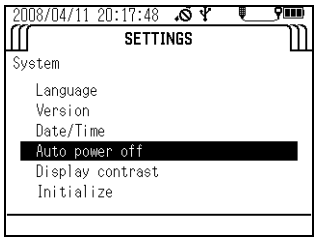
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

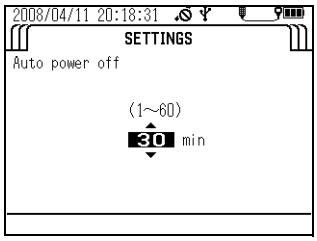
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**



4. **Press the down (▽) key to move the cursor to "Auto power off", then press the ENTER key.**



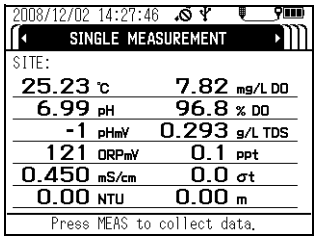
5. **Press the up (△) and down (▽) keys to select the desired time setting, then press the ENTER key.**
   You can select OFF, or settings of 1, 2, 5, 10, 20, 30 or 60 minutes.
   Default: 30 minutes



● Display contrast

Follow the steps below to adjust the display's contrast.

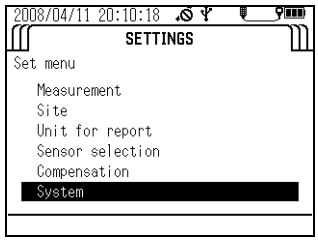
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.



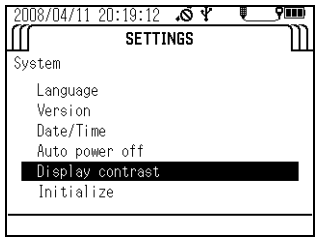
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

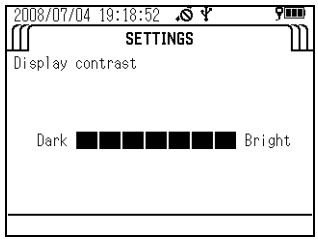
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**



4. **Press the down (▽) key to move the cursor to "Display contrast", then press the ENTER key.**



5. **Press the left (◁) and right (▷) keys to adjust the contrast.**
   Adjustment can be made in 26 steps.

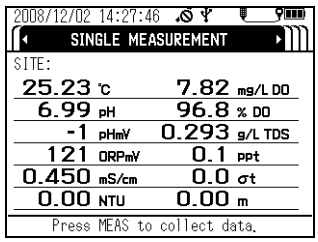


6. **Press the ENTER key.**

● Initialization

Follow the steps below to restore all the settings except date/time to their factory defaults. Factory default calibration data for the electrical conductivity and turbidity sensors will also be deleted at the same time.

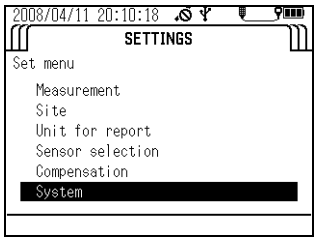
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.



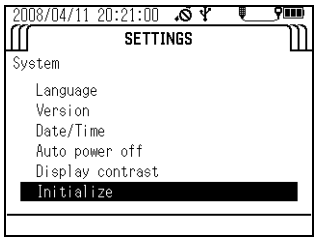
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

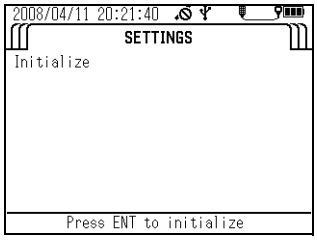
2. **Press the right (▷) key to switch the display to the "SETTINGS" screen.**
3. **Press the down (▽) key to move the cursor to "System", then press the ENTER key.**



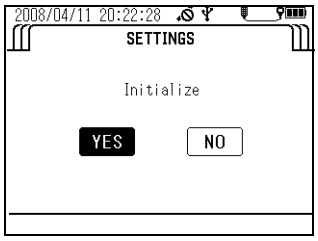
4. **Press the down (▽) key to move the cursor to "Initialize", then press the ENTER key.**



5. **Press the ENTER key again.**



6. **A confirmation message appears asking whether to execute initialization. Press the left (◁) key to move the cursor to YES, then press the ENTER key.**
   The message "Initialize Complete" appears to indicate the process has finished.



3.3 Calibration

To obtain correct measurement values, the sensors need to be calibrated using standard solution before measurement. You can select simultaneous auto calibration of the pH, COND and TURB sensors in pH4 standard solution and DO and DEP sensors simultaneously in air, or manual calibration of individual measurement parameters. You can check the result of the previous calibration using the procedure on " 3.5.4 Checking the calibration record " (page 70).

Note

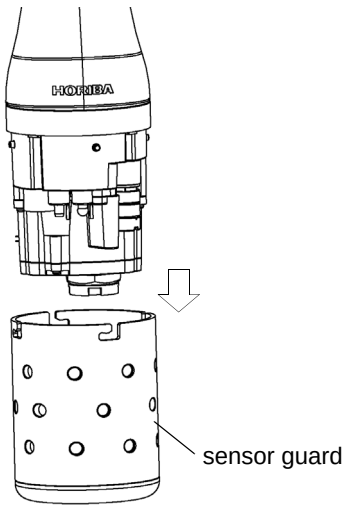
- **Wait at least 20 minutes after turning the system power ON before calibrating the DO sensor.**
- **Make the DO and COND compensation settings before calibration since these settings are applied during calibration.**
- **You can select only the desired parameters for calibration and calibrate just those parameters (see " 3.2.4 Sensor selection " (page 25)).**
- **Use about 200 mL of standard solution in the calibration cup.**
- **Calibration data is stored in the sensor probe.**

3.3.1 Auto calibration

Tip

- **The following parameters are calibrated (at 25°C):**
  pH: Set to 4.01 (zero-point calibration); the span is adjusted to the factory default value.
  COND: 0.449 S/m (4.49 mS/cm, span calibration); the zero point is adjusted to the factory default value.
  TURB: 0 NTU (zero-point calibration); the span is adjusted to the factory default value.
  DO: 8.92 mg/L (span calibration); the zero point is adjusted to the factory default value.
  DEP: 0 m (zero-point calibration); the zero point is adjusted to the factory default value.
- **If the air temperature changes, the readout value may not be stable. Ensure that the ambient air temperature is the same temperature as the calibration solution, because the internal probe temperature sensor and external temperature sensor (in the calibration solution) are used for the auto calibration. Allow the probe and standard solution to equilibrate for 1 hour if a thermometer is not available to verify that these temperatures are the same.**
- **Do not hold the probe while performing the auto calibration. Body temperature may elevate the internal temperature sensor measurement creating DO calibration error.**

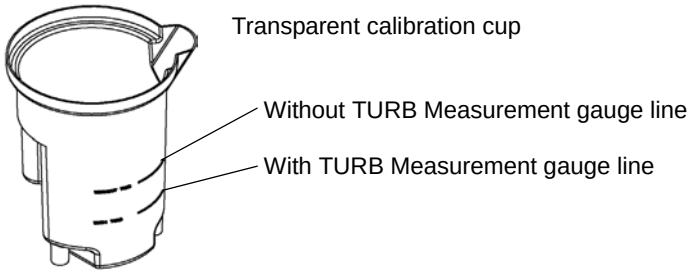
1. **Remove the sensor guard and wash the sensor probe 2 or 3 times with deionized water.**



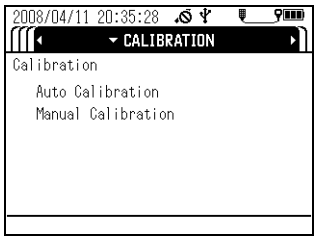
2. **Remove the transparent calibration cup.**

3. **Fill the transparent calibration cup to the line with pH 4 standard solution.**

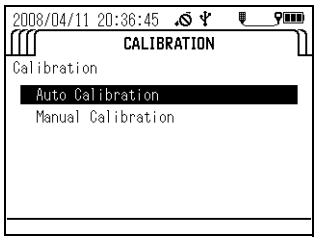
The transparent calibration cup has With TURB Measurement and Without TURB Measurement gauge lines.



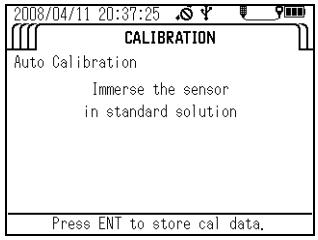
4. **Press the control unit's CAL key to set the calibration mode.**

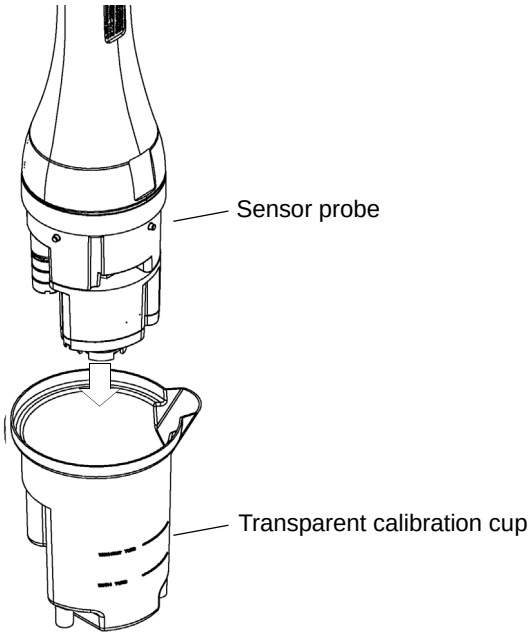


5. **Press the down (▽) key to move the cursor to "Auto Calibration", then press the ENTER key.**

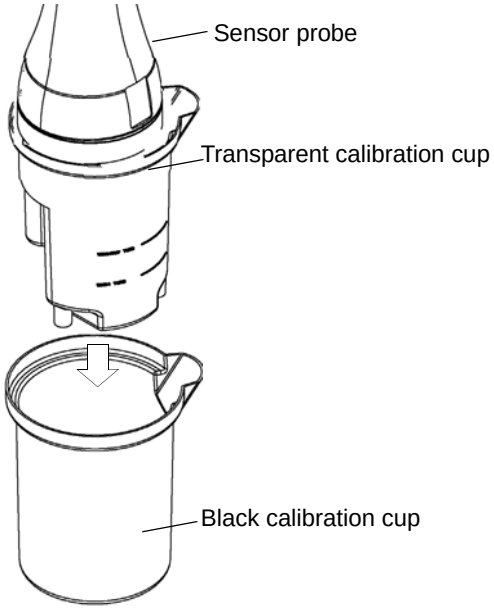


6. **Immerse the sensor probe in the transparent calibration cup.**
Check that the pH sensor, ORP sensor, reference electrode, COND sensor, TURB sensor and temperature sensor are submerged in the pH 4 standard solution and check that there are no air bubbles on the sensor.





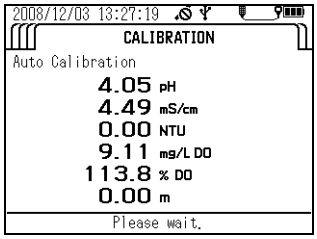
7. **With the sensor probe still in the transparent calibration cup, place the transparent calibration cup into the black calibration cup.**



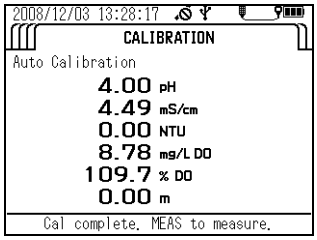
8. **When all the sensor values have stabilized, press the ENTER key to start calibration.**

Note

Do not remove the sensor probe from the calibration solution. U-53 turbidity data will display "----" until the calibration is completed.



Calibration is finished when the message "Cal complete. MEAS to measure." appears. Press the MEAS key to set the measurement screen, then start measurement.



If a calibration error occurs, start calibration after first resolving the issue according to the instructions in " 4.6 Troubleshooting " (page 89).

3.3.2 Manual calibration

The procedures below describe how to calibrate each sensor individually.

Note

The displayed units are the units set by selecting "Unit for report" in the "SETTINGS" screen.

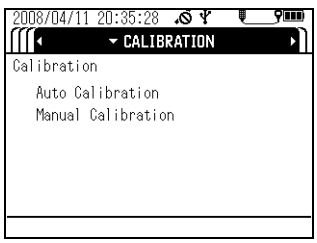
- **Temperature (TEMP) calibration**

1. **Fill a bucket or similar container with water of a known temperature, and insert the sensor probe in it.**

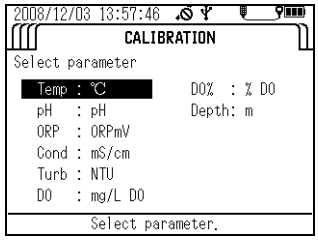
Wait 5 minutes before starting calibration to allow the sensor probe temperature to stabilize.

2. **Press the control unit's CAL key to set the calibration mode.**

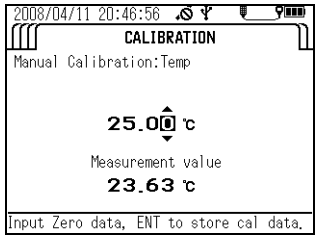
3. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



4. **In the parameter selection screen, move the cursor to “Temp”, then press the ENTER key.**



5. **Press the up (△) and down (▽) keys to set the calibration value - the temperature of the water containing the submerged sensor probe.**



6. **Check that “Measurement value” has stabilized, then press the ENTER key to start calibration.**
Calibration is finished when the message “Cal complete. CNT to measure.” appears.

● pH calibration

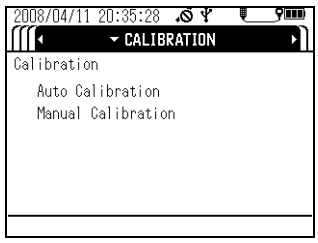
Note

You can select one calibration point (zero-point calibration) or two calibration points (zero-point calibration and span calibration). Carry out two calibration procedures to ensure good measurement precision throughout all measurement ranges.

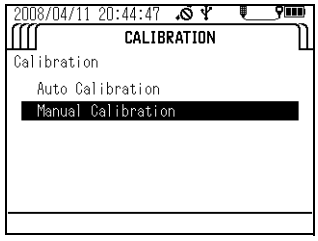
1. **Calibrate the zero point. Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with pH 7 standard solution.**

2. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

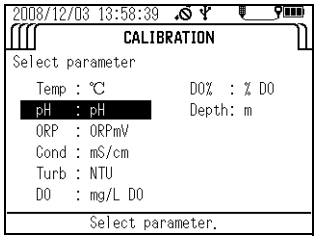
3. **Press the control unit’s CAL key to set the calibration mode.**



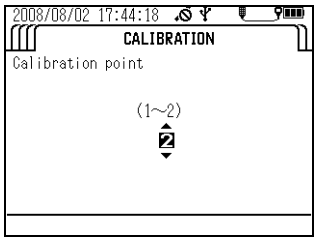
4. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



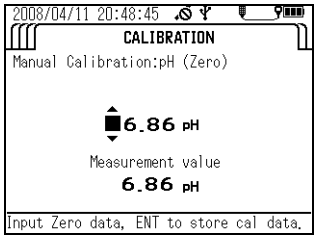
5. **In the parameter selection screen, move the cursor to "pH", then press the ENTER key.**



6. **Set the number of calibration points, then press the ENTER key.**



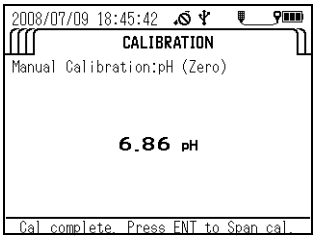
7. **Press the up (△) and down (▽) keys to set the pH value of the pH 7 standard solution containing the submerged sensor probe at the measurement temperature**



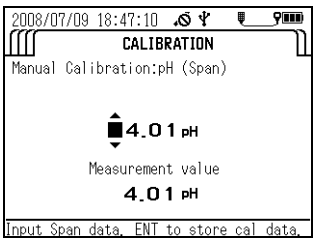
Temp. (°C)	pH 4 standard solution Phthalate	pH 7 standard solution Neutral phosphate	pH 9 standard solution Borate
0	4.01	6.98	9.46
5	4.01	6.95	9.39
10	4.00	6.92	9.33
15	4.00	6.90	9.27
20	4.00	6.88	9.22
25	4.01	6.86	9.18
30	4.01	6.85	9.14
35	4.02	6.84	9.10
40	4.03	6.84	9.07
45	4.04	6.84	9.04

8. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

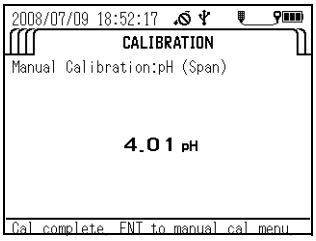
9. **Press the ENTER key to start the span calibration procedure when the message "Cal complete. Press ENT to Span cal." appears.**



10. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with pH 4 or pH 9 standard solution.**
11. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**
12. **Press the up (△) and down (▽) keys to set the pH value of the pH 4 or pH 9 standard solution containing the submerged sensor probe at the measurement temperature.**



13. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**
14. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter**

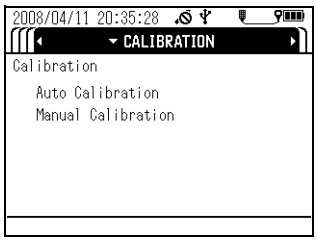


● ORP calibration

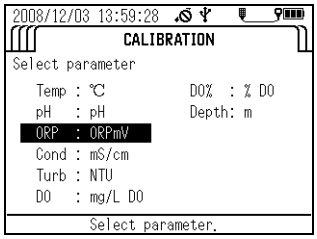
Note

- **If the prepared ORP standard solution is left in open air for one hour or more, the solution may be transformed. For this reason ORP standard solution cannot be stored.**
  Calibrate within one hour of preparing the solution.
- **When measuring sample with low concentrations of oxidants and reductants after conducting an operational check using a standard substance, the measured values may not stabilize or the results of measurement might not be repeatable. If this is the case, start the measurement after immersing the sensors in the sample water sufficiently.**
- **Note that when measuring the ORP of solution with extremely low concentrations of oxidants and reductants, such as tap water, well water, or water treated with purifying equipment, there may be less responsiveness, repeatability, and stability, in general.**
- **When alkaline ion water is left for 5 minutes, its ORP undergoes changes significantly. Always measure alkaline ion water promptly.**

1. **Fill a clean beaker with one bag of ORP standard powder No. 160-22 or No. 160-51. Add 250 mL of deionized water and agitate the solution thoroughly (there will be some excess quinhydrone (a black powder) that floats on the surface when agitating the solution). Fill the transparent calibration cup to the reference line with this standard solution.**
2. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**
3. **Press the control unit's CAL key to set the calibration mode.**
4. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



5. **In the parameter selection screen, move the cursor to ORP, then press the ENTER key.**



6. **Press the up (△) and down (▽) keys to set the mV value of the ORP standard solution containing the submerged sensor probe at the measurement temperature.**

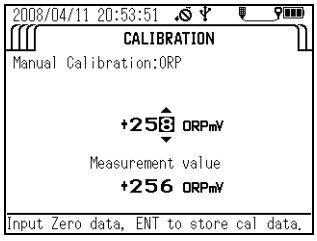


Table 1 Indicated value of ORP standard solution at various temperatures (mV)

Temperature	160-22	16051
5	+274	+112
10	+271	+107
15	+267	+101
20	+263	+95
25	+258	+89
30	+254	+83
35	+249	+76
40	+244	+69

7. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

8. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter selection screen.**

● Conductivity (COND) calibration

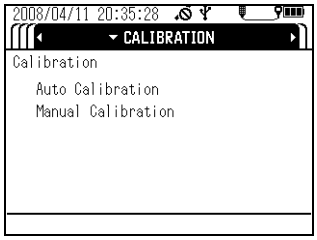
Note

- **To support a wide range of sample concentrations, electrical conductivity is divided into three measurement ranges: 0.0 mS/m to 99.9 mS/m, 0.090 S/m to 0.999 S/m, and 0.9 S/m to 9.99 S/m.**
- **When manually calibrating conductivity, you can select two calibration points (one zero-point calibration point and a span calibration point for one of the three measurement ranges) or four calibration points (one zero-point calibration point and span calibration points for all three measurement ranges). Carry out the four calibration points to ensure good measurement precision throughout all measurement ranges.**
- **Make the compensation setting before calibration since this setting is applied during calibration. (Refer to " 6.5.3 Temperature coefficient " (page 104)).**

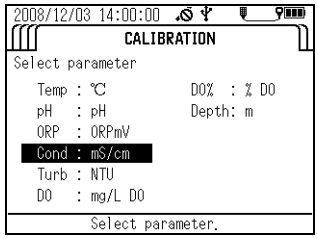
1. **Prepare the standard solution. Dry Potassium chloride (KCl) powder (high-grade commercially available) at 105°C for two hours, and leave it to cool in a desiccator.**
2. **Consult the following table and weigh potassium chloride (KCl), then prepare three standard potassium chloride (KCl) solutions following the procedure below.**

Potassium chloride (KCl) standard solution	Conductivity (COND) value	Potassium chloride (KCl) mass (g) at solution temperature of 25 °C	Calibration range
0.005 mol/L	71.8 mS/m (0.718 mS/cm)	0.373	0.0 mS/m to 99.9 mS/m (0.00 mS/cm to 0.999 mS/cm)
0.050 mol/L	0.667 S/m (6.67 mS/cm)	3.73	0.090 S/m to 0.999 S/m (1.00 mS/cm to 9.99 mS/cm)
0.500 mol/L	5.87 S/m (58.7 mS/cm)	37.2	0.9 S/m to 9.99 S/m (10.0 mS/cm to 99.9 mS/cm)

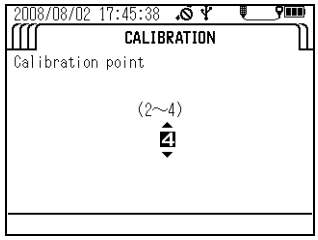
3. **Dissolve the weighed Potassium Chloride (KCl) in deionized water.**
4. **Put the dissolved Potassium Chloride (KCl) into a 1 L measuring flask, and fill to the 1 L mark with deionized water.**
5. **Calibrate the zero point. Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then remove all moisture from the sensor probe (it will be calibrated in air).**
6. **Press the control unit's CAL key to set the calibration mode.**
7. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



8. **In the parameter selection screen, move the cursor to “Cond”, then press the ENTER key.**

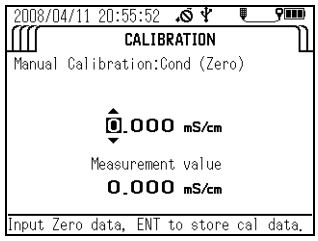


9. **Set the number of calibration points, then press the ENTER key.**

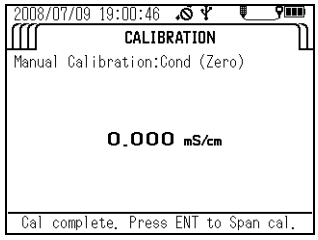


The instructions below assume that four calibration points have been set.

10. **Press the up (△) and down (▽) keys to set the "Cond" value to 0.0 mS/m (0.000 mS/cm).**
11. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

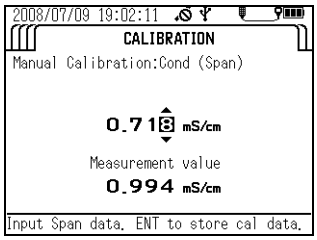


12. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the first span calibration procedure.**



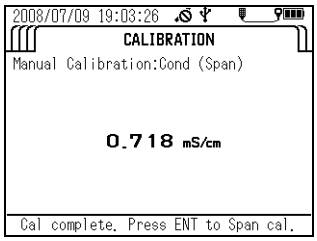
13. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 71.8 mS/m (0.718 mS/cm) standard solution.**
14. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

15. **Press the up (△) and down (▽) keys to set the "Cond" value to 71.8 mS/m (0.718 mS/cm).**
Calibration range = 0 mS/m to 99.9 mS/m (0 mS/cm to 0.999 mS/cm)



16. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

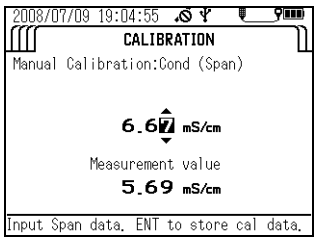
17. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the next span calibration procedure.**



18. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 0.667 S/m (6.67 mS/cm) standard solution.**

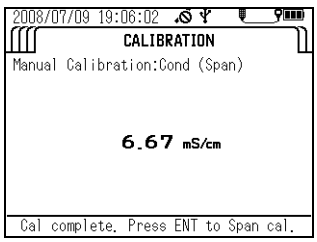
19. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

20. **Press the up (△) and down (▽) keys to set the "Cond" value to 0.667 S/m (6.67 mS/cm).**
Calibration range = 0.100 S/m to 0.999 S/m (1.00 mS/cm to 9.99 mS/cm)



21. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

22. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the next span calibration procedure.**

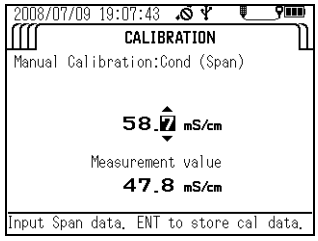


23. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 5.87 S/m (58.7 mS/cm) standard solution.**

24. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

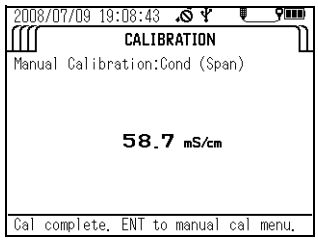
25. **Press the up (△) and down (▽) keys to set the "Cond" value to 5.87 S/m (58.7 mS/cm).**

Calibration range = 1.00 S/m to 10.00 S/m(10.0 mS/cm to 100.0 mS/cm)



26. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

27. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter selection screen.**



● Turbidity (TURB) calibration

Note

- **To support a wide range of sample concentrations, turbidity is divided into three measurement ranges: 0.0 to 9.9 NTU, 10 to 100 NTU, and over 100 NTU.**
- **When manually calibrating turbidity, you can select two calibration procedures (one zero-point calibration procedure and a span calibration procedure for one of the three measurement ranges), three calibration procedures (one zero-point calibration procedure and a span calibration procedure for two of the three measurement ranges) or four calibration procedures (one zero-point calibration procedure and span calibration procedures for all three measurement ranges). Carry out the four calibration procedures to ensure good measurement precision throughout all measurement ranges.**
- **Always use the calibration cup provided. Using other containers can create effects from ambient light that cause incorrect calibration.**

● Preparing the standard solutions

1. **Weigh out 5.0 g of hydrazine sulfate (commercial special grade or above), and dissolve it in 400 mL of deionized water. Dissolve 50 g of hexamethylene tetramine (commercial special grade or above) in 400 mL of deionized water in anther flask.**
2. **Mix the two solutions and add deionized water until the total solution volume is 1000 mL, and mix well. Store this solution at a temperature of 25°C ±3°C for 48 hours.**
   The turbidity value (TURB) of this solution is equivalent to 4000 NTU.
3. **Dilute 4000 NTU-solution 5 times (use a pipette to measure 50 mL of the 4000 NTU solution and pour it into a 250 mL measuring flask, and fill up to 250 mL meniscus)**
   The turbidity value (TURB) of this solution is equivalent to 800 NTU.
4. **Dilute 800 NTU solution 10 times (use a pipette to measure 25 mL of the 800 NTU solution and pour it into a 250 mL measuring flask, and fill up to 250 mL meniscus)**
   The turbidity value (TURB) of this solution is equivalent to 80 NTU.
5. **Dilute 80 NTU solution 10 times (use a pipette to measure 25 mL of the 80 NTU solution and pour it into a 250 mL measuring flask, and fill up to 250 mL meniscus)**
   The turbidity value (TURB) of this solution is equivalent to 8 NTU.

Note

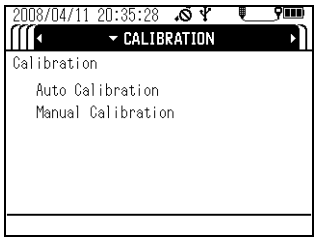
Instead of the standard solutions above, you can use other standard solutions of known concentration measured with other standard instruments.

● U-52, U-53 turbidity calibration

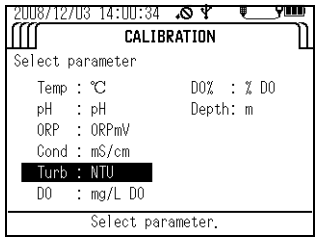
Set the number of calibration points.

You can set between 2 and 4 points.

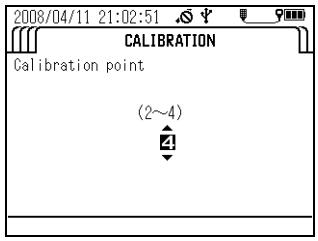
1. **Press the control unit's CAL key to set the calibration mode.**
2. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



3. **In the parameter selection screen, move the cursor to "Turb", then press the ENTER key.**



4. **Press the up (△) and down (▽) keys to set the number of calibration points, then press the ENTER key.**

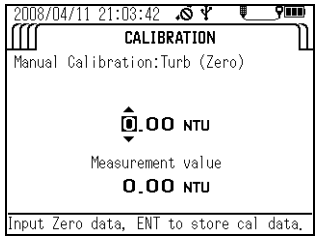


The instructions below assume that four calibration points have been set.

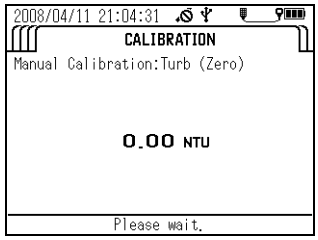
5. **Calibrate the zero point. Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with deionized water.**

6. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

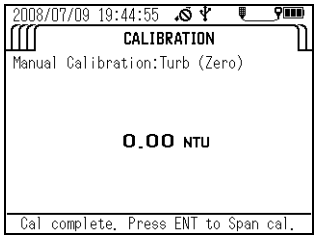
7. **Press the up (△) and down (▽) keys to set the "Turb" value to 0.0 NTU.**



8. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**



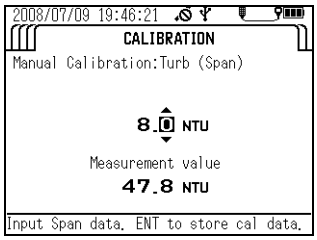
9. When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the first span calibration procedure.



10. Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 8 NTU standard solution, or a standard solution of known concentration between 0.1 and 10 NTU.

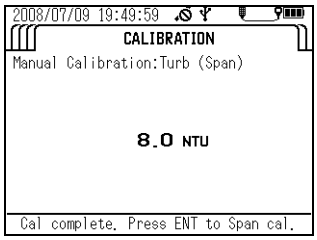
11. Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.

12. Press the up (△) and down (▽) keys to set the "TURB" value to 8 NTU, or to the known concentration of the standard solution between 0.1 and 10 NTU. (Input range = 0 NTU to 9.9 NTU (U-51) or 0 NTU to 9.99 NTU (U-52))



13. Check that "Current measurement value" has stabilized, then press the ENTER key to start calibration.

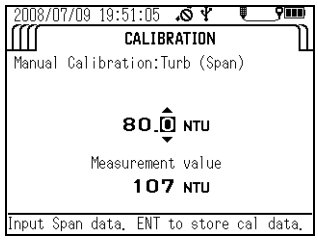
14. When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the next span calibration procedure.



15. Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 80 NTU standard solution, or a standard solution of known concentration between 10 and 100 NTU.

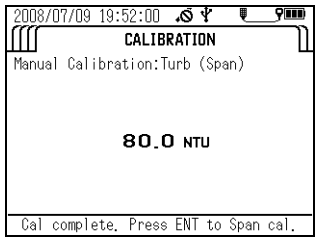
16. Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.

17. **Press the up (△) and down (▽) keys to set the "Turb" value to 80 NTU, or to the known concentration of the standard solution between 10 and 100 NTU. (Input range = 10.0 NTU to 99.9 NTU)**



18. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

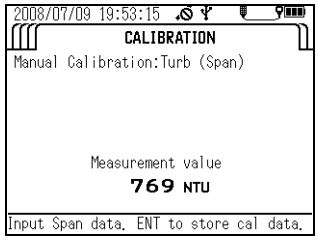
19. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the next span calibration procedure.**



20. **Wash the transparent calibration cup 2 or 3 times with deionized water, then fill it to the reference line with 800 NTU standard solution, or a standard solution of known concentration 100 NTU above.**

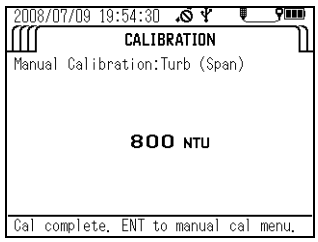
21. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the transparent calibration cup.**

22. **Press the up (△) and down (▽) keys to set the "TURB" value to 800 NTU, or to the known concentration of the standard solution 100 NTU above.**
(Input range = 100 NTU to 800 NTU (U-51), 100 NTU to 1000 NTU (U-52))



23. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**

24. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter selection screen.**



● Dissolved oxygen (DO) calibration

Note

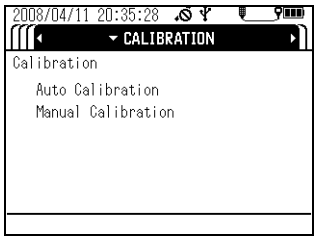
- **You can select one calibration procedure (span calibration) or two calibration procedures (zero-point calibration and span calibration). Carry out the two calibration procedures to ensure good measurement precision throughout all measurement ranges.**
- **It is necessary to prepare new solution before calibration of the Dissolved Oxygen (DO) sensor.**
- **The calibration cup (included) cannot be used to manually calibrate the DO sensor. Use a suitable bottle in which the DO sensor and the temperature sensor can be immersed.**
- **Wait at least 20 minutes after turning the system power ON before calibrating the DO sensor.**
- **Make the compensation setting before calibration since the setting is applied during calibration.**
- **The DO sensor is affected by flow. When performing span calibration with saturated dissolved oxygen water, move the cable slowly up and down (move the sensor probe at a rate of roughly 20 to 30 cm a second) or agitate the saturated dissolved oxygen water.**

1. **Prepare the standard solution.**

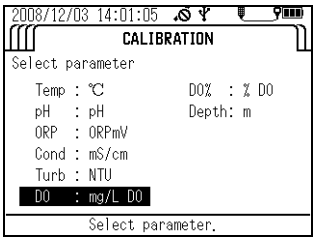
- Add about 50 g of sodium sulfite to 1000 mL of water (either deionized water or tap water) and stir the mixture to dissolve the sodium sulfite in it.
- Pour 1 to 2 liters of water into a suitable flask (either deionized water or tap water). Using a air pump, feed air into the water and aerate the solution until oxygen is saturated.

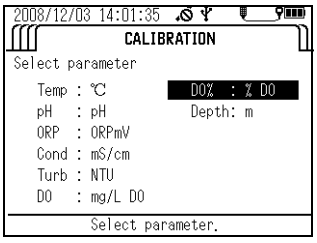
2. **First, calibrate the zero point. Press the control unit's CAL key to set the calibration mode.**

3. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**

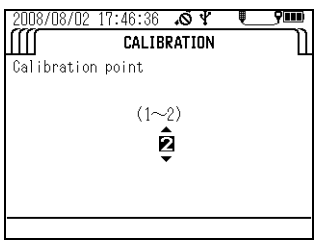


4. **In the parameter selection screen, move the cursor to DO or DO%, then press the ENTER key.**



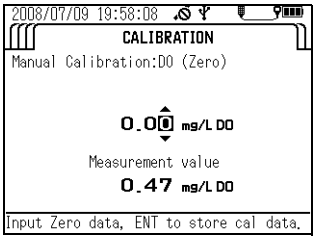


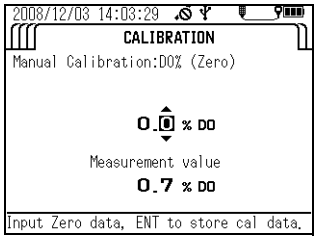
5. **Set the number of calibration procedures, then press the ENTER key.**



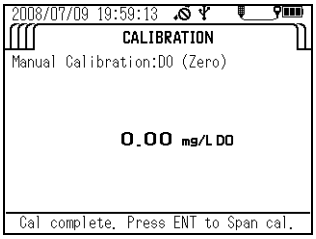
The instructions below assume that two calibration points have been set.

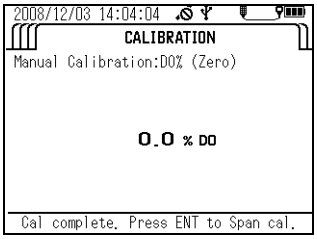
6. **Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then submerge the sensor probe in the bottle.**
7. **Press the up (△) and down (▽) keys to set the DO value to 0.00 mg/L or 0.0%.**



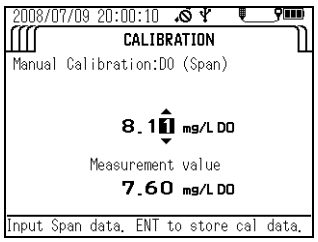


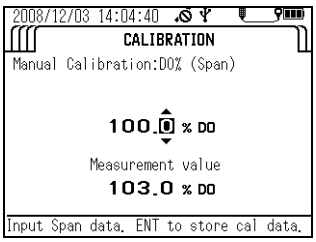
8. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**
9. **When the message "Cal complete. Press ENT to Span cal." appears, press the ENTER key to start the span calibration procedure.**



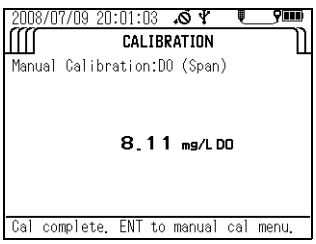


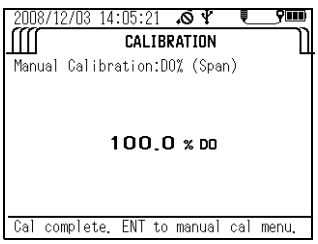
10. **Wash the sensor probe 2 or 3 times with deionized water to remove any dirt, then submerge the sensor probe in the container filled with the span solution.**
11. **Press the up (△) and down (▽) keys to set the DO value to the saturated dissolved oxygen value (mg/L) of the water at that temperature or the dissolved oxygen saturation ratio.**





12. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**
13. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter selection screen.**





Amounts of saturated dissolved oxygen in water at various temperatures (salinity=0.0%)

JIS K0101

Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)
0	14.16						
1	13.77	11	10.67	21	8.68	31	7.42
2	13.40	12	10.43	22	8.53	32	7.32
3	13.04	13	10.20	23	8.39	33	7.22
4	12.70	14	9.97	24	8.25	34	7.13
5	12.37	15	9.76	25	8.11	35	7.04
6	12.06	16	9.56	26	7.99	36	6.94
7	11.75	17	9.37	27	7.87	37	6.86
8	11.47	18	9.18	28	7.75	38	6.76
9	11.19	19	9.01	29	7.64	39	6.68
10	10.92	20	8.84	30	7.53	40	6.59

ISO5814

Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)
0	14.62				
1	14.22	11	11.03	21	8.91
2	13.83	12	10.78	22	8.74
3	13.46	13	10.54	23	8.58
4	13.11	14	10.31	24	8.42
5	12.77	15	10.08	25	8.26
6	12.45	16	9.87	26	8.11
7	12.14	17	9.66	27	7.97
8	11.84	18	9.47	28	7.83
9	11.56	19	9.28	29	7.69
10	11.29	20	9.09	30	7.56

● **Span setting values for calibration in air**

The software should display these values when auto calibration is performed.
Use this table to input values for manual span calibrations in air.

Tip

The DO measurement value of "air-saturated water" and air are different.
Due to the pressure difference against the membrane in air versus the membrane in water, the measurement value in air is about 10% higher than the value of air-saturated water on average.

Amounts of saturated dissolved oxygen in air at various temperatures

Following tables are applicable only to the air calibration of the U-50 DO sensor. Do not use them for other purpose.

Air calibration value in adopting evaluation based on JIS K0101

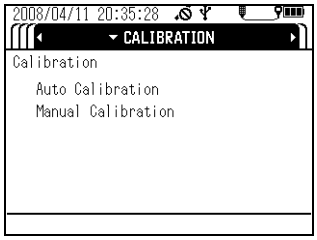
Temp (°C)	DO (mg/L)	Temp (°C)	DO (mg/L)	Temp (°C)	DO (mg/L)	Temp (°C)	DO (mg/L)
0	15.58						
1	15.15	11	11.74	21	9.55	31	8.16
2	14.74	12	11.47	22	9.38	32	8.05
3	14.34	13	11.22	23	9.23	33	7.94
4	13.97	14	10.97	24	9.08	34	7.84
5	13.61	15	10.74	25	8.92	35	7.74
6	13.27	16	10.52	26	8.79	36	7.63
7	12.93	17	10.31	27	8.66	37	7.55
8	12.62	18	10.10	28	8.53	38	7.44
9	12.31	19	9.91	29	8.40	39	7.35
10	12.01	20	9.72	30	8.28	40	7.25

Air calibration value in adopting evaluation based on ISO5814

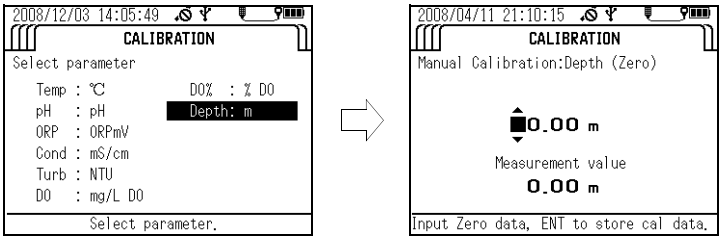
Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)	Temp. (°C)	DO (mg/L)
0	16.08				
1	15.64	11	12.13	21	9.80
2	15.21	12	11.86	22	9.61
3	14.81	13	11.59	23	9.44
4	14.42	14	11.34	24	9.26
5	14.05	15	11.09	25	9.09
6	13.70	16	10.86	26	8.92
7	13.35	17	10.63	27	8.77
8	13.02	18	10.42	28	8.61
9	12.72	19	10.21	29	8.46
10	12.42	20	10.00	30	8.32

● Water depth (DEPTH) calibration

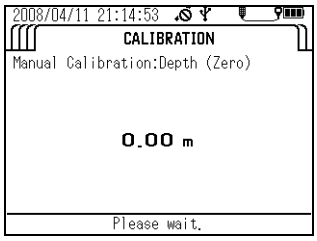
1. **Calibrate the zero point. Wash the sensor probe 2 or 3 times in deionized water to remove any dirt, then remove all moisture from the sensor probe (it will be calibrated in air).**
2. **Press the control unit's CAL key to set the calibration mode.**
3. **Press the down (▽) key to move the cursor to "Manual Calibration", then press the ENTER key.**



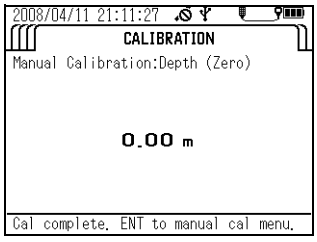
4. **In the parameter selection screen, move the cursor to "Depth", then press the ENTER key.**



5. **Press the up (△) and down (▽) keys to set the "Depth" value to 0.00 m.**
6. **Check that "Measurement value" has stabilized, then press the ENTER key to start calibration.**



7. **Calibration is finished when the message "Cal complete. ENT to manual cal menu." appears. Press the ENTER key to return to the calibration parameter selection screen.**



3.4 Measurement

You can perform measurement by either of the methods below.

- Storing data in memory manually with reference to the measurement value (single measurement)
- Having data stored in memory automatically and continuously
  U-51/U-52: Interval measurement (minimum memory interval of 10 seconds)
  U-53: Interval measurement (minimum memory interval of 30 seconds)

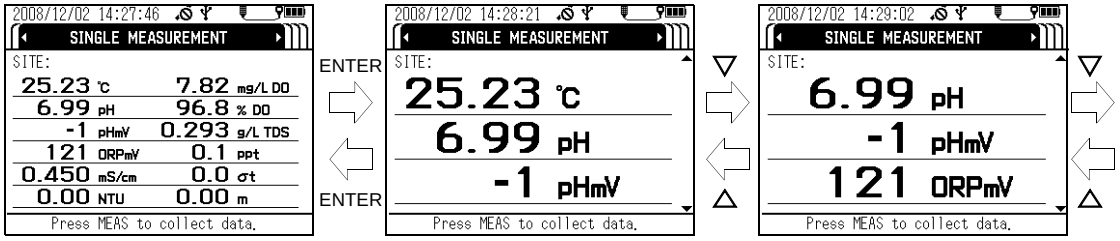
Select the measurement method that meets your requirements.

Note

- **Lower sensor probe slowly when submerging them in samples.**
- **Sensors may break if sensor probe are dropped from a height of 1 meter or more.**
- **Do not submerge sensor probe in water depths of over 30 meters. Sensor probe are only resistant to water pressure of up to 30 meters.**
- **After turning the power ON, check that the DO readout value has stabilized before starting measurement (takes around 20 minutes).**

Tip

- **When on the measurement screen, pressing the ENTER key enlarges the display and shows three measured values at a time.**
- **Pressing the up (△) and down (▽) keys scrolls through the measured values one item at a time.**
- **Pressing the ENTER key again reverts to the normal measurement screen display.**



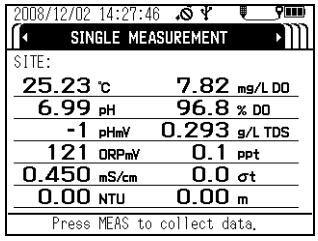
Measurement screen Normal Enlarged

3.4.1 Storing data in memory manually

Follow the steps below to manually store data in memory while referring to the measurement value to check the readout value is stable.

● **U-51/U-52**

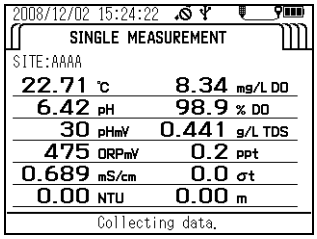
1. **Check that each sensor and sensor guard is mounted.**
2. **Check that "SINGLE MEASUREMENT" has been selected in the measurement screen.**



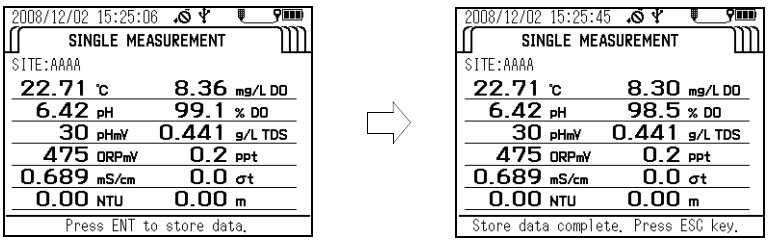
3. **Submerge the sensor probe in the sample, gently shaking them in the sample to remove any air bubbles from the sensors.**

If the sample is non-flowing, move the cable slowly up and down (move the sensor probe at a rate of roughly 20 to 30 cm a second) to ensure that fresh sample is continuously supplied to the DO sensor.

4. **When the measurement values are stable, press the MEAS key to acquire the 5-second average.**



5. **Press the ENTER key to save the held measurement values, or press the ESC key to cancel the operation.**

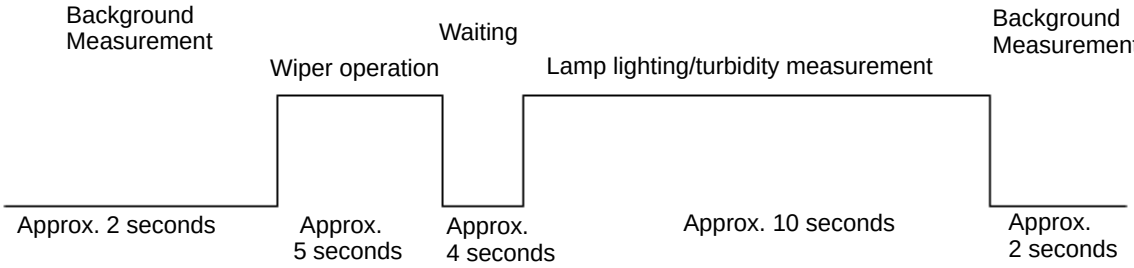


● U-53

Note

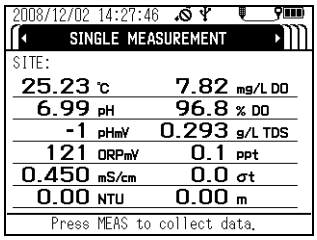
Do not perform turbidity measurement in air as it may damage the wiper.

U-53 turbidity measurement follows the sequence below. The measurement values are held after each sequence.

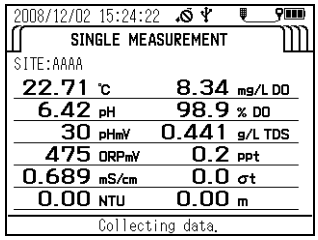


1. **Check that each sensor and sensor guard is mounted.**

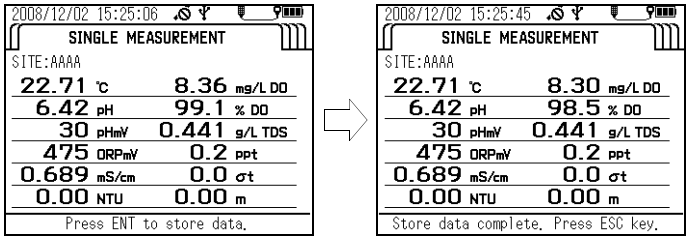
2. **Check that "SINGLE MEASUREMENT" has been selected in the measurement screen.**



3. **Submerge the sensor probe in the sample, gently shaking them in the sample to remove any air bubbles from the sensors.**
   If the sample is non-flowing, move the cable slowly up and down (move the sensor probe at a rate of roughly 20 to 30 cm a second) to ensure that fresh sample is continuously supplied to the DO sensor.
4. **When the non-turbidity meter measurement values are stable, press the MEAS key to start the sequence above.**



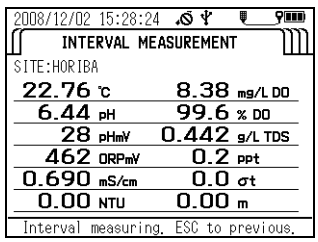
5. **When the sequence has finished, hold the measurement values. Press the ENTER key to store the held measurement values, or press the ESC key to cancel the operation.**



3.4.2 Automatic, continuous measurement

● **Interval measurement**

1. **Select the "Interval measurement" measurement setting (see " 3.2.1 Setting measurement methods " (page 18)).**
2. **Press the up (△) and down (▽) keys to set the interval value to the desired value (U-51/U-52: minimum interval: 10 seconds, U-53: minimum interval: 30 seconds), then press the ENTER key.**
   The measurement screen appears automatically, and the system becomes ready for measurement.
3. **Check that each sensor and sensor guard is mounted.**
4. **Submerge the sensor probe in the sample, gently shaking them in the sample to remove any air bubbles from the sensors.**
   If the sample is non-flowing, move the cable slowly up and down (move the sensor probe at a rate of roughly 20 to 30 cm a second) to ensure that fresh sample is continuously supplied to the DO sensor.
5. **Press the ENTER key to start measurement.**



3.5 Data operations

Use the procedures below to retrieve data stored in memory, delete all the data, check the remaining data memory capacity, and check the calibration record.

3.5.1 Displaying data

For maximum efficiency, there are 3 methods of displaying data.

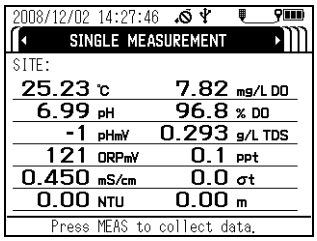
- Displaying the data for a specified site
- Displaying the data for a specified date/time
- Displaying all the data

Use the method that best suits your requirements.

● **Displaying the data for a specified site**

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

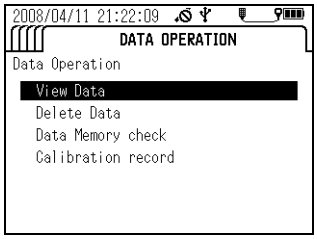


Note

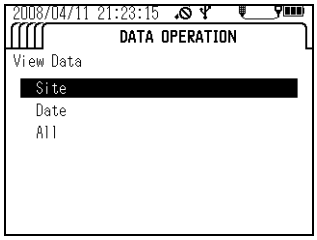
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**

3. **Press the down (▽) key to move the cursor to "View Data", then press the ENTER key.**

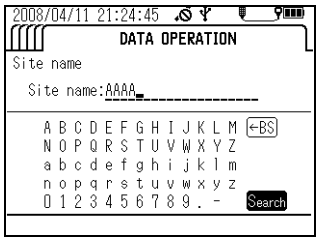


4. **Move the cursor to "Site", then press the ENTER key.**



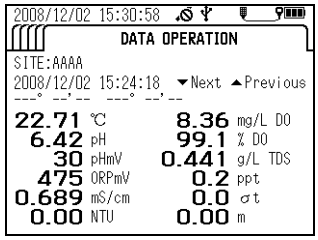
5. **Press the up (△), down (▽), left (◁) and right (▷) keys to enter the site to retrieve.**

6. **Move the cursor to "Search", then press the ENTER key.**



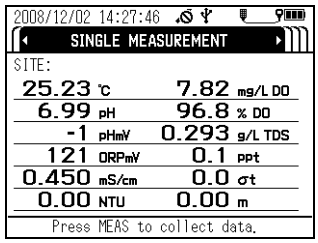
All site names that begin with the entered text are displayed.
The most recently measured data for the entered site is displayed.

7. **Press the up (△) and down (▽) keys to display earlier data.**



● Displaying the data for a specified date/time

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

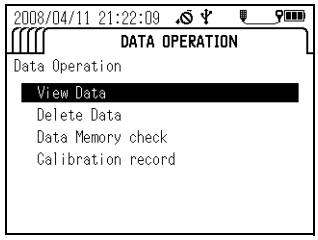


Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

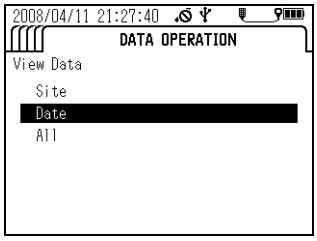
2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**

3. **Press the down (▽) key to move the cursor to "View Data", then press the ENTER key.**



4. **Move the cursor to "Date", then press the ENTER key.**

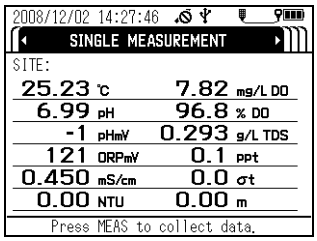
5. **With the cursor on the Date, press the ENTER key.**



6. **Press the up (△), down (▽), left (◁) and right (▷) keys to enter the desired date/ time, then press the ENTER key to apply the setting.**
7. **The cursor moves to "Search". Press the ENTER key to start the search.**
8. **Press the up (△) and down (▽) keys to display earlier data.**

● **Displaying all the data**

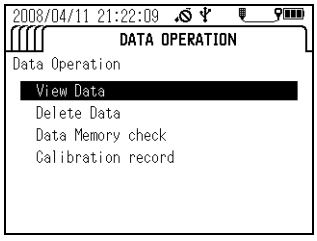
1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.



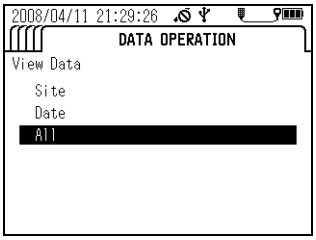
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**
3. **Press the down (▽) key to move the cursor to "View Data", then press the ENTER key.**



4. **Move the cursor to "All", then press the ENTER key.**
The most recently measured data is displayed.

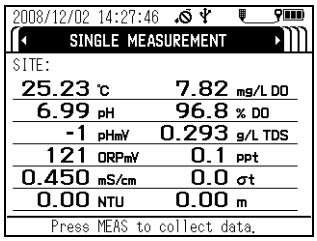


5. **Press the up (△) and down (▽) keys to display earlier data.**

3.5.2 Deleting data

Follow the steps below to delete all the data stored in memory.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
The "MEASUREMENT" screen appears after about 10 seconds.

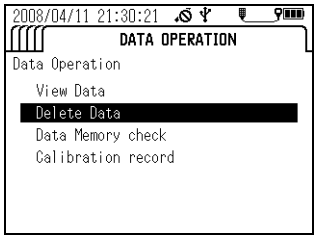


Note

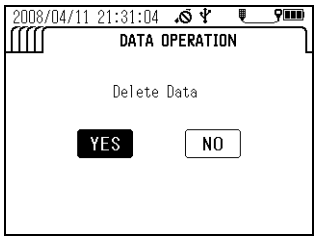
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**

3. **Press the down (▽) key to move the cursor to "Delete Data", then press the ENTER key.**



4. **Press the left (◁) key to move the cursor to YES, then press the ENTER key.**
All the data has been deleted when the indicator appears along with the message "No data exists".

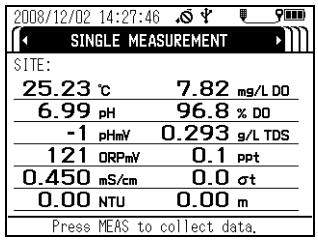


3.5.3 Checking the data memory

You can check the used data capacity and the remaining data capacity.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.

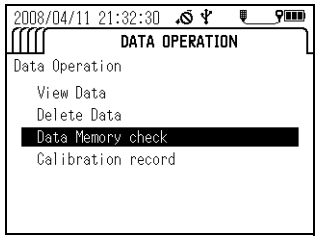


Note

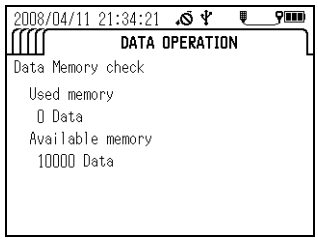
The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**

3. **Press the down (▽) key to move the cursor to "Data Memory Check", then press the ENTER key.**



The amount of memory in use and amount of available memory are displayed.

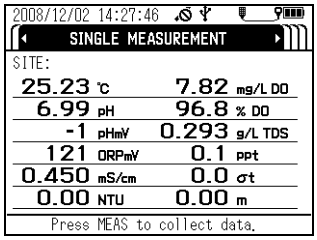


3.5.4 Checking the calibration record

Follow the steps below to check the latest calibration history.

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**

The "MEASUREMENT" screen appears after about 10 seconds.



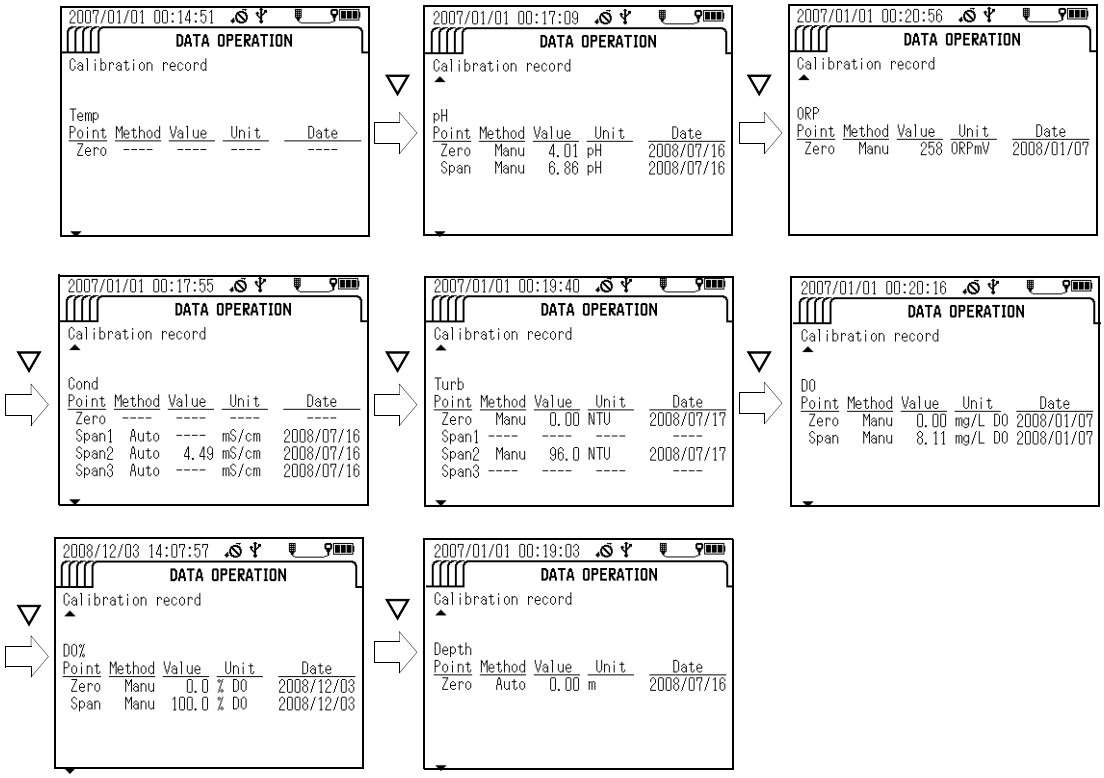
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Press the right (▷) key 3 times to display the "DATA OPERATION" screen.**

3. **Press the down (▽) key to move the cursor to "Calibration record", then press the ENTER key.**

The latest calibration record is displayed.



3.5.5 GPS data operations

The menu for GPS data operations appears on the display to which the GPS unit is mounted.

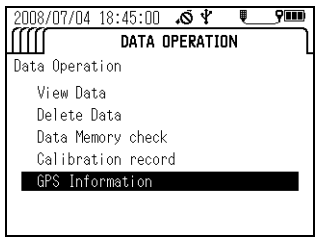
● GPS information

Follow the steps below to display acquired GPS information.

Note

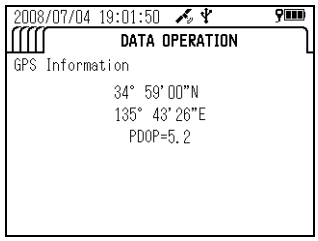
Turning the power OFF erases the GPS information.

1. **Press the right (▷) key to switch the display to the "DATA OPERATION" screen.**
2. **the down (▽) key to move the cursor to "GPS Information", then press the ENTER key.**

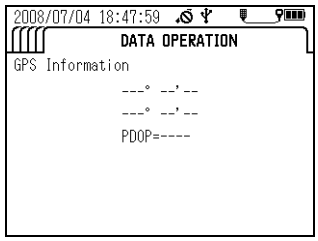


The last GPS information acquired is displayed.

● When received data exists

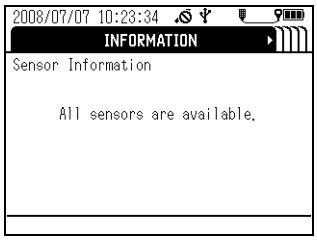


● When no received data exists

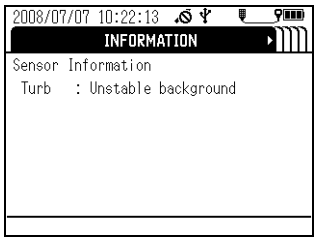


3.6 Sensor information

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power ON.**
   The "MEASUREMENT" screen appears after about 10 seconds.
2. **Press the left (◁) key once to display the "INFORMATION" screen.**
   The "Sensor Information" screen displays the sensor probe's status.
   ● When the sensor probe is normal, the display below appears.



   ● When there is a sensor probe problem, individual measurement parameters generate messages such as the one shown below. Follow the troubleshooting information to remove the problem before continuing to operate the system.

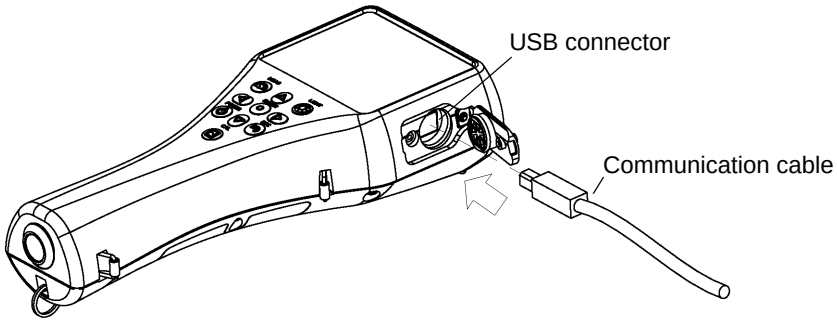


3.7 USB communication

The separately-sold, dedicated PC connection cable comes with data collection software. This software allows data to be downloaded from the control unit in CSV format.

This section contains instructions for communication commands used for USB communication.

● Connecting the cable



Dedicated cable

Part name: Communication cable (with data collection software)

Part no.: 3200174823

● Cautions when using USB communication

Take care to observe the following when using USB communication.

- Use the dedicated cable (with data collection software) or a commercially-available USB cable (A-B type) to connect to a PC.
- Be sure to match the transmission format on the control unit and the computer.
  The control unit uses the following transmission format:

Baud rate:	19200 bps
Number of stop bits:	1 bit
Data bit length:	8 bits
Parity:	None
Flow control:	None

Tip

If the transmission formats do not match, a communication error occurs and USB communication will not function normally. After changing the transmission format, restart the control unit and the computer.

- If received data is not sent back or an error occurs after a data request has been sent, adjust the program configuration so that it allows a little waiting time before a data request is sent again. This will enable more stable communication.
- The unit does not use DCD, CTS, or DSR signals. Take care of this when creating programs.

3.7.1 Communication settings

Baud rate:	19200 bps
Number of stop bits:	1 bit
Data bit length:	8 bits
Parity:	None
Flow control:	None

3.7.2 Commands

● Instant data requests

● Request command format

#	RD	@	XX	[CR]	[LF]
1	2	3	4		

1	Header	1 character
2	Command	2 characters
3	Delimiter character	1 character
4	Frame check sequence (FCS)	2 characters

The two ASCII-code characters created by converting the 8 bits of data created by successively combining the value of each character from # through @ in an exclusive OR (XOR) operation with the value of the next character.

Example: #RD@

(1)	0	XOR	35	(ASCII code of # symbol)	⇒	35
(2)	35	XOR	82	(ASCII code of R)	⇒	113
(3)	113	XOR	68	(ASCII code of D)	⇒	53
(4)	53	XOR	64	(ASCII code of @ symbol)	⇒	117 (decimal) ↓ 75 (hex) ↓ Sets "75".

Example: 35 XOR 82 operation

35 in binary	⇒	0	0	1	0	0	0	1	1	
82 in binary	⇒	0	1	0	1	0	0	1	0	
XOR result		0	1	1	1	0	0	0	1	⇒ 113 (decimal)

Note: Set "XX" if you do not want to test for communication frame errors with FCS.

● Response format

#	RD	AAAAAAAAAAAAAAAAAAAA	X	X	XXXX	XX	X	X	XXXXX	X
1	2	3	4	5	6	7	8	9	10	11

XX	X	X	XXXXX	X	XX	X	X	XXXXX	X	XX	X	X	XXXXX	X
12	13	14	15	16	17	18	19	20	21	22	23	24	25	26

XX	X	X	XXXXX	X	XX	X	X	XXXXX	X	XX	X	X	XXXXX	X
27	28	29	30	31	32	33	34	35	36	37	38	39	40	41

XX	X	X	XXXXX	X	XX	X	X	XXXXX	X	XX	X	X	XXXXX	X
42	43	44	45	46	47	48	49	50	51	52	53	54	55	56

XX	X	X	XXXXX	X	XX	X	X	XXXXX	X	XX	X	X	XXXXX	X
57	58	59	60	61	62	63	64	65	66	67	68	69	70	71

XX	XX	XX	XX	XX	XX	XX	XX	XX	X	X	XXX	XX	XX	X	X	@	XX	[CR]	[LF]
72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89		

1	Header		1 character
2	Command		2 characters
3	Site name	Upper- and lowercase letters, numbers, periods (.) hyphens (-) and spaces ( )	20 characters
4	Probe status	(3) Status code	1 character
5	Probe error	(4) Status error code	1 character
6	Unused		4 characters
7	Parameter 1 code	(1) Parameter code	2 characters
8	Parameter 1 status	(5) Parameter status code	1 character
9	Parameter 1 error	(6) Parameter error code	1 character
10	Parameter 1 data	5 characters including decimal point, right-justified with blanks filled	5 characters
11	Parameter 1 unit	(2) Unit code	1 character
12	Parameter 2 code	(1) Parameter code	2 characters
13	Parameter 2 status	(5) Parameter status code	1 character
14	Parameter 2 error	(6) Parameter error code	1 character
15	Parameter 2 data	5 characters including decimal point, right-justified with blanks filled	5 characters
16	Parameter 2 unit	(2) Unit code	1 character
17	Parameter 3 code	(1) Parameter code	2 characters
18	Parameter 3 status	(5) Parameter status code	1 character
19	Parameter 3 error	(6) Parameter error code	1 character
20	Parameter 3 data	5 characters including decimal point, right-justified with blanks filled	5 characters
21	Parameter 3 unit	(2) Unit code	1 character
22	Parameter 4 code	(1) Parameter code	2 characters
23	Parameter 4 status	(5) Parameter status code	1 character
24	Parameter 4 error	(6) Parameter error code	1 character
25	Parameter 4 data	5 characters including decimal point, right-justified with blanks filled	5 characters
26	Parameter 4 unit	(2) Unit code	1 character
27	Parameter 5 code	(1) Parameter code	2 characters
28	Parameter 5 status	(5) Parameter status code	1 character
29	Parameter 5 error	(6) Parameter error code	1 character
30	Parameter 5 data	5 characters including decimal point, right-justified with blanks filled	5 characters
31	Parameter 5 unit	(2) Unit code	1 character
32	Parameter 6 code	(1) Parameter code	2 characters
33	Parameter 6 status	(5) Parameter status code	1 character
34	Parameter 6 error	(6) Parameter error code	1 character

35	Parameter 6 data	5 characters including decimal point, right-justified with blanks filled	5 characters
36	Parameter 6 unit	(2) Unit code	1 character
37	Parameter 7 code	(1) Parameter code	2 characters
38	Parameter 7 status	(5) Parameter status code	1 character
39	Parameter 7 error	(6) Parameter error code	1 character
40	Parameter 7 data	5 characters including decimal point, right-justified with blanks filled	5 characters
41	Parameter 7 unit	(2) Unit code	1 character
42	Parameter 8 code	(1) Parameter code	2 characters
43	Parameter 8 status	(5) Parameter status code	1 character
44	Parameter 8 error	(6) Parameter error code	1 character
45	Parameter 8 data	5 characters including decimal point, right-justified with blanks filled	5 characters
46	Parameter 8 unit	(2) Unit code	1 character
47	Parameter 9 code	(1) Parameter code	2 characters
48	Parameter 9 status	(5) Parameter status code	1 character
49	Parameter 9 error	(6) Parameter error code	1 character
50	Parameter 9 data	5 characters including decimal point, right-justified with blanks filled	5 characters
51	Parameter 9 unit	(2) Unit code	1 character
52	Parameter 10 code	(1) Parameter code	2 characters
53	Parameter 10 status	(5) Parameter status code	1 character
54	Parameter 10 error	(6) Parameter error code	1 character
55	Parameter 10 data	5 characters including decimal point, right-justified with blanks filled	5 characters
56	Parameter 10 unit	(2) Unit code	1 character
57	Parameter 11 code	(1) Parameter code	2 characters
58	Parameter 11 status	(5) Parameter status code	1 character
59	Parameter 11 error	(6) Parameter error code	1 character
60	Parameter 11 data	5 characters including decimal point, right-justified with blanks filled	5 characters
61	Parameter 11 unit	(2) Unit code	1 character
62	Parameter 12 code	(1) Parameter code	2 characters
63	Parameter 12 status	(5) Parameter status code	1 character
64	Parameter 12 error	(6) Parameter error code	1 character
65	Parameter 12 data	5 characters including decimal point, right-justified with blanks filled	5 characters
66	Parameter 12 unit	(2) Unit code (6) Parameter error code	1 character
67	Parameter 13 code	(1) Parameter code	2 characters
68	Parameter 13 status	(5) Parameter status code	1 character
69	Parameter 13 error	(6) Parameter error code	1 character
70	Parameter 13 data	5 characters including decimal point, right-justified with blanks filled	5 characters
71	Parameter 13 unit	(2) Unit code	1 character
72	Year	00 to 99	2 characters

73	Month	01 to 12	2 characters
74	Day	01 to 31	2 characters
75	Hour	00 to 23	2 characters
76	Minute	00 to 59	2 characters
77	Second	00 to 59	2 characters
78	Longitude (degrees)	00 to 90 or "--" (no GPS data)	2 characters
79	Longitude (minutes)	00 to 59 or "--" (no GPS data)	2 characters
80	Longitude (seconds)	00 to 59 or "--" (no GPS data)	2 characters
81	Unused	1 character	1 character
82	North latitude/South latitude	N: North; S: South	1 character
83	Latitude (degrees)	000 to 180 or "---" (no GPS data)	3 characters
84	Latitude (minutes)	00 to 59 or "--" (no GPS data)	2 characters
85	Latitude (seconds)	00 to 59 or "--" (no GPS data)	2 characters
86	Unused		1 character
87	East longitude/West longitude	E: East; W: West	1 character
88	Delimiter character		1 character
89	Frame check sequence (FCS)		2 characters

● Memory data requests

● Request command format

#	RM	X	X	AAAAAAAAAAAAAAAAAAAA	XX	XX	XX	@	XX	[CR]	[LF]
1	2	3	4	5	6	7	8	9	10		

1	Header		1 character
2	Command		2 characters
3	Data specification[*1]	0: Start search; 1: Next data item; 2: Previous data item; 3: Request same data again	1 character
4	Search method specification	0: All data; 1: Site search; 2: Date search	1 character
5	Search site[*2]	Upper- and lowercase letters, numbers, periods (.) hyphens (-) and spaces ( )	20 characters
6	Search year[*3]	00 to 99	2 characters
7	Search month[*3]	01 to 12	2 characters
8	Search day[*3]	01 to 31	2 characters
9	Delimiter character		1 character
10	Frame check sequence (FCS)		2 characters

*1: When sending the RM command, first send 0 [Start search], then 1 [Next data item], 2 [Previous data item] or 3 [Request same data again].

*2: [Search site] is only needed when [Site search] is specified as the search method. If another search method is specified, fill this field with spaces.

*3: [Search year], [Search month] and [Search day] are only needed when [Date search] is specified as the search method. If another search method is specified, fill this field with spaces.

● **Response format**

(when data exists)

```
#   RM  AAAAAAAAAAAAAAAAAAAA   XX  X   X   XXXXX  X
1   2   3                      4   5   6   7      8

XX  X   X   XXXXX  X   XX  X   X   XXXXX  X   XX  X   X   XXXXX  X
9   10  11  12      13  14  15  16  17      18  19  20  21  22      23

XX  X   X   XXXXX  X   XX  X   X   XXXXX  X   XX  X   X   XXXXX  X
24  25  26  27      28  29  30  31  32      33  34  35  36  37      38

XX  X   X   XXXXX  X   XX  X   X   XXXXX  X   XX  X   X   XXXXX  X
39  40  41  42      43  44  45  46  47      48  49  50  51  52      53

XX  X   X   XXXXX  X   XX  X   X   XXXXX  X   XX  X   X   XXXXX  X
54  55  56  57      58  59  60  61  62      63  64  65  66  67      68

XX  XX  XX  XX  XX  XX  XX  XX  XX  X   X   XXX  XX  XX  X   X   @   XX  [CR]  [LF]
69  70  71  72  73  74  75  76  77  78  79  80   81  82  83  84  85  86
```

1	Header		1 character
2	Command		2 characters
3	Site name	Upper- and lowercase letters, numbers, periods (.) hyphens (-) and spaces ( )	20 characters
4	Parameter 1 code	(1) Parameter code	2 characters
5	Parameter 1 selection	0: No selection; 1: Selection made	1 character
6	Parameter 1 error	(6) Parameter error code	1 character
7	Parameter 1 data	5 characters including decimal point, right-justified with blanks filled	5 characters
8	Parameter 1 unit	(2) Unit code	1 character
9	Parameter 2 code	(1) Parameter code	2 characters
10	Parameter 2 selection	0: No selection; 1: Selection made	1 character
11	Parameter 2 error	(6) Parameter error code	1 character
12	Parameter 2 data	5 characters including decimal point, right-justified with blanks filled	5 characters
13	Parameter 2 unit	(2) Unit code	1 character
14	Parameter 3 code	(1) Parameter code	2 characters
15	Parameter 3 selection	0: No selection; 1: Selection made	1 character
16	Parameter 3 error	(6) Parameter error code	1 character
17	Parameter 3 data	5 characters including decimal point, right-justified with blanks filled	5 characters
18	Parameter 3 unit	(2) Unit code	1 character
19	Parameter 4 code	(1) Parameter code	2 characters
20	Parameter 4 selection	0: No selection; 1: Selection made	1 character

21	Parameter 4 error	(6) Parameter error code	1 character
22	Parameter 4 data	5 characters including decimal point, right-justified with blanks filled	5 characters
23	Parameter 4 unit	(2) Unit code	1 character
24	Parameter 5 code	(1) Parameter code	2 characters
25	Parameter 5 selection	0: No selection; 1: Selection made	1 character
26	Parameter 5 error	(6) Parameter error code	1 character
27	Parameter 5 data	5 characters including decimal point, right-justified with blanks filled	5 characters
28	Parameter 5 unit	(2) Unit code	1 character
29	Parameter 6 code	(1) Parameter code	2 characters
30	Parameter 6 selection	0: No selection; 1: Selection made	1 character
31	Parameter 6 error	(6) Parameter error code	1 character
32	Parameter 6 data	5 characters including decimal point, right-justified with blanks filled	5 characters
33	Parameter 6 unit	(2) Unit code	1 character
34	Parameter 7 code	(1) Parameter code	2 characters
35	Parameter 7 selection	0: No selection; 1: Selection made	1 character
36	Parameter 7 error	(6) Parameter error code	1 character
37	Parameter 7 data	5 characters including decimal point, right-justified with blanks filled	5 characters
38	Parameter 7 unit	(2) Unit code	1 character
39	Parameter 8 code	(1) Parameter code	2 characters
40	Parameter 8 selection	0: No selection; 1: Selection made	1 character
41	Parameter 8 error	(6) Parameter error code	1 character
42	Parameter 8 data	5 characters including decimal point, right-justified with blanks filled	5 characters
43	Parameter 8 unit	(2) Unit code	1 character
44	Parameter 9 code	(1) Parameter code	2 characters
45	Parameter 9 selection	0: No selection; 1: Selection made	1 character
46	Parameter 9 error	(6) Parameter error code	1 character
47	Parameter 9 data	5 characters including decimal point, right-justified with blanks filled	5 characters
48	Parameter 9 unit	(2) Unit code	1 character
49	Parameter 10 code	(1) Parameter code	2 characters
50	Parameter 10 selection	0: No selection; 1: Selection made	1 character
51	Parameter 10 error	(6) Parameter error code	1 character
52	Parameter 10 data	5 characters including decimal point, right-justified with blanks filled	5 characters
53	Parameter 10 unit	(2) Unit code	1 character
54	Parameter 11 code	(1) Parameter code	2 characters
55	Parameter 11 selection	0: No selection; 1: Selection made	1 character
56	Parameter 11 error	(6) Parameter error code	1 character
57	Parameter 11 data	5 characters including decimal point, right-justified with blanks filled	5 characters
58	Parameter 11 unit	(2) Unit code	1 character
59	Parameter 12 code	(1) Parameter code	2 characters

60	Parameter 12 selection	0: No selection; 1: Selection made	1 character
61	Parameter 12 error	(6) Parameter error code	1 character
62	Parameter 12 data	5 characters including decimal point, right-justified with blanks filled	5 characters
63	Parameter 12 unit	(2) Unit code	1 character
64	Parameter 13 code	(1) Parameter code	2 characters
65	Parameter 13 selection	0: No selection; 1: Selection made	1 character
66	Parameter 13 error	(6) Parameter error code	1 character
67	Parameter 13 data	5 characters including decimal point, right-justified with blanks filled	5 characters
68	Parameter 13 unit	(2) Unit code	1 character
69	Year	00 to 99	2 characters
70	Month	01 to 12	2 characters
71	Day	01 to 31	2 characters
72	Hour	00 to 23	2 characters
73	Minute	00 to 59	2 characters
74	Second	00 to 5	2 characters
75	Longitude (degrees)	00 to 90 or "--" (no GPS data)	2 characters
76	Longitude (minutes)	00 to 59 or "--" (no GPS data)	2 characters
77	Longitude (seconds)	00 to 59 or "--" (no GPS data)	2 characters
78	Unused		1 character
79	North latitude/South latitude	N: North; S: South	1 character
80	Latitude (degrees)	000 to 180 or "---" (no GPS data)	3 characters
81	Latitude (minutes)	00 to 59 or "--" (no GPS data)	2 characters
82	Latitude (seconds)	00 to 59 or "--" (no GPS data)	2 characters
83	Unused		1 character
84	East longitude/West longitude	E: East; W: West	1 character
85	Delimiter character		1 character
86	Frame check sequence (FCS)		2 characters

When no data exists, or memory is at capacity)

#	RM	@	XX	[CR]	[LF]
1	2	3	4		

1	Header	1 character
2	Command	2 characters
3	Delimiter character\	1 character
4	Frame check sequence (FCS)	2 characters

● Memory data count request

● Request command format

#	RN	@	XX	[CR]	[LF]
1	2	3	4		

1	Header	1 character
2	Command	2 characters
3	Delimiter character\	1 character
4	Frame check sequence (FCS)	2 characters

● Response format

#	RN	XXXXX	@	XX	[CR]	[LF]
1	2	3	4	5		

1	Header		1 character
2	Command		2 characters
3	Total data count	0 to 10000	5 characters
4	Delimiter character\		1 character
5	Frame check sequence (FCS)		2 characters

● Command parse failure response

#	??	X	XX	X	@	XX	[CR]	[LF]
1	2	3	4	5	6	7		

1	Header	1 character
2	Command	2 characters
3	Command parse failure reason*4	1 character
4	Received command*5	2 characters
5	(3) Status code for probe status*5	1 character
6	Delimiter character	1 character
7	Frame check sequence (FCS)	2 characters

*4: List of command parse failure reasons

1: Frame length error
2: FCS mismatch
3: Undefined command
4: Data error
5: Data out of range
6: No "@" delimiter character
7: No "#" header character
8: No [Carriage return] + [Line feed] footer
9: Cannot accept command in this timing.

*5: Only set for command parse failure reason 9, [Cannot accept command in this timing]. Otherwise this field is filled with spaces.

4 Maintenance

Tip

HORIBA recommends regular manufacturer maintenance checks in order to ensure a long product life.

4.1 Routine care

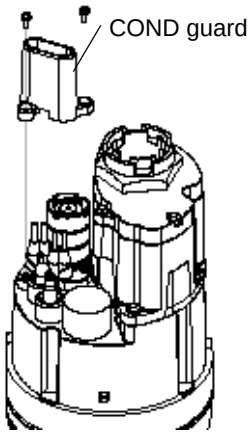
- **After measurement**

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power OFF.**

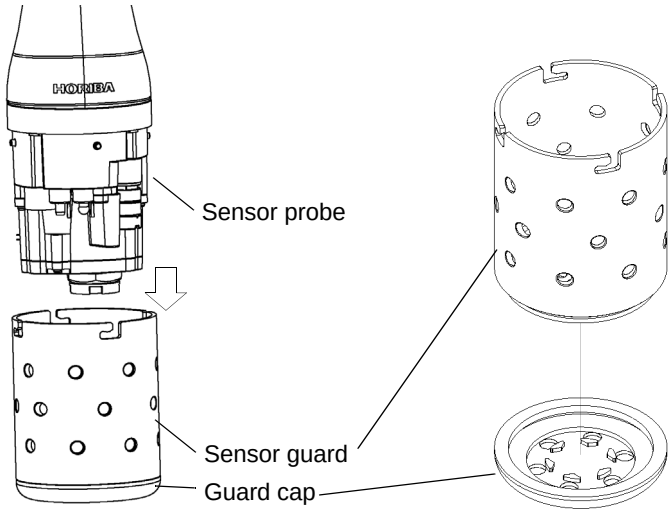
Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Remove the sensor guard, and clean the sensor with tap water.**
3. **Clean the turbidity sensor with the cleaning brush provided.**
4. **Remove the two screws securing the COND guard, and the COND guard itself, and use a test tube brush to gently remove any dirt from the electrical conductivity electrode.**



5. **Wipe off any dirt with a soft cloth. If parts are very dirty, clean them with neutral detergent, then rinse them. If parts are contaminated by oil, wipe it off with a soft cloth soaked in alcohol.**
6. **Put the COND guard back in place.**
7. **Remove the sensor guard's guard cap, wash off any dirt with tap water, then put the guard cap back in place.**



4.2 Every 2 months maintenance

● Dissolved oxygen (DO) sensor

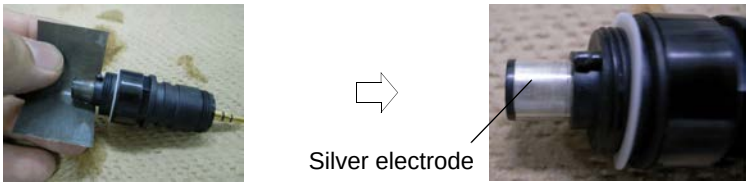
Note

- **The DO sensor's internal solution is potassium chloride (KCl). Although KCl is harmless, protective equipment such as gloves and goggles should be worn when working with it.**
- **Internal solution can be disposed of down a sink.**

- Replace the membrane cap.
- Polish the gold and silver electrodes when replacing the membrane cap.
  The gold electrode does not need to be polished if it is not dirty.

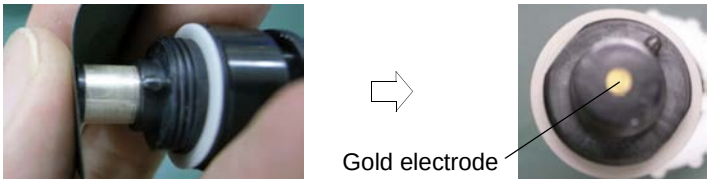
● **Silver electrode**

Polish a silver electrode part with sandpaper (#500) and then wash metal electrode parts with water.



● **Gold electrode**

Polish a gold electrode part with sandpaper (#8000) and then wash metal electrode parts with water.



Replace a membrane cap after clean metal electrodes parts.
Refer to “ 4.5 Replacing the membrane cap ” (page 87).

● **Reference electrode**

Note

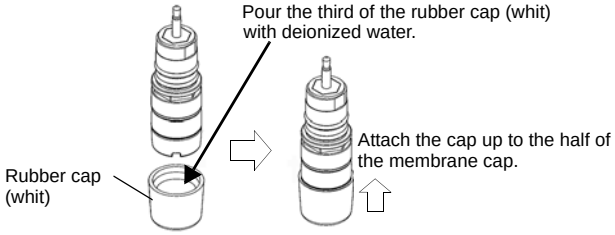
- **The pH reference internal solution is potassium chloride (KCl). Although KCl is harmless, protective equipment such as gloves and goggles should be worn when working with it.**
- **Internal solution can be disposed of down a sink.**

1. **Remove the rubber liquid junction plug from the reference electrode and dispose of the internal solution.**
2. **To prevent air entering, fill the reference electrode to the brim with its internal solution (No. 330).**
3. **Put the rubber liquid junction plug back in place.**
   If the rubber liquid junction plug is dirty, replace the liquid junctions (set of two; No. 9037005100). The reference electrode's internal solution will spill when replacing the liquid junctions. Rinse parts with tap water and dry them with a soft cloth.

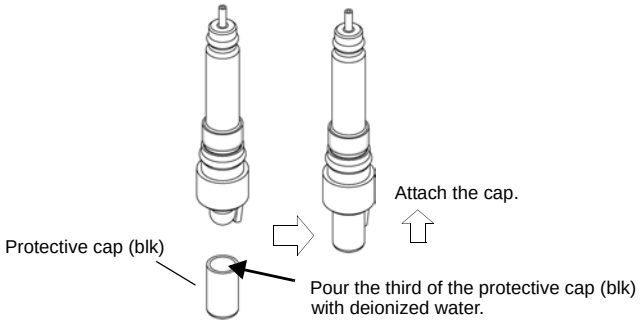
4.3 Storage

● Short-term (under 2 months) storage

- Before storing the DO sensor, pour the third of the rubber cap (whit) provided with deionized water and cover the DO sensor with them.



- Before storing the pH sensor, pour the third of the protective cap (blk) provided with deionized water and cover the pH sensor with them.



Note

Before measuerment, remove the rubber cap (whit) and the protective cap (blk).

● Long-term (2 months or more) storage

- Remove a membrane cap from DO sensor, and wash the gold electrode and silver electrode parts with water. Wipe off the moisture before storing DO sensor in the pack.
- Prevent internal solution seeping out of the reference chip by taping over the point of seepage with electrical tape.
- Before storing the system, remove the control unit's batteries to prevent battery leakage.

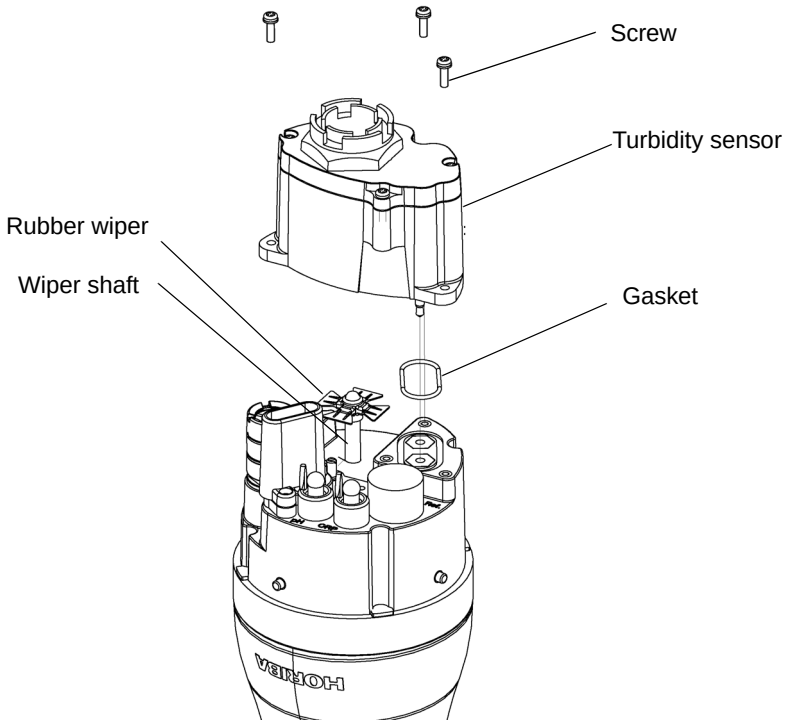
4.4 Replacing the turbidity sensor

1. **Press and hold down the control unit's POWER key for about 3 seconds to turn the power OFF.**

Note

The operation keys are designed to operate using the pad of a finger, sharp objects can tear the control unit cover damaging the operation keys.

2. **Remove the sensor guard, and clean the sensor probe with tap water.**
3. **Use dry air to blow away and dry off any moisture.**
4. **Remove the three screws holding the turbidity sensor by using No. 2 Phillips head screwdriver.**
5. **Pull out the turbidity sensor horizontally.**
6. **Remove the rubber wiper and gasket, and use a soft cloth to wipe off any dirt from the wiper shaft and turbidity sensor attachment. If parts are very dirty, use a soft cloth soaked in neutral detergent or alcohol.**
7. **Replace the rubber wiper and gasket with new ones. Coat the gasket with a thin layer of grease (No. 3014017718).**
8. **Attach the new turbidity sensor and fasten it in place with the three screws.**
9. **Perform four-point calibration before using the sensor.**



4.5 Replacing the membrane cap

● Replacement procedure

1. **Prepare the DO sensor.**

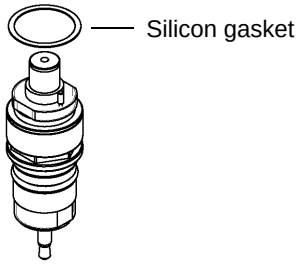
- Take a DO sensor out of pack (newly purchasing).
- Remove a DO sensor from the sensor probe (after use).

Newly purchasing

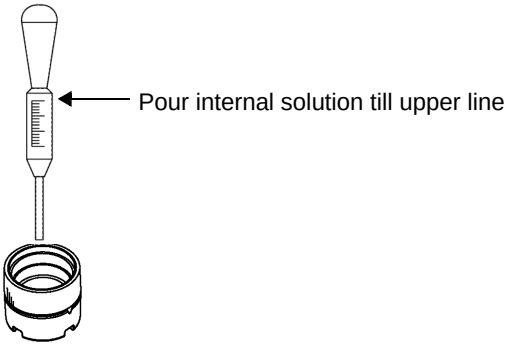
Undo a DO sensor from the sensor probe

- Twist a membrane cap from DO sensor.
- Wash the gold electrode and silver electrode parts with water.

2. **Replace the silicone gasket with a new one.**



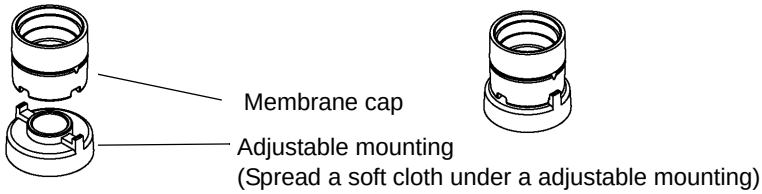
3. **Pour internal solution into a membrane cap with a dropper.**



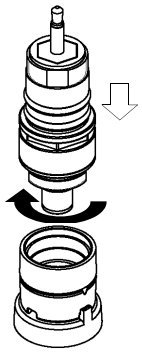
- Check air bubbles in a membrane cap.

Pick a Cap up and drop it down, if there is air bubbles in internal solution of it.

4. **Set up a membrane cap on a adjustable mounting.**



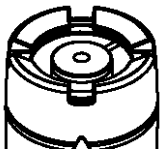
5. **Attach a membrane cap to DO sensor**



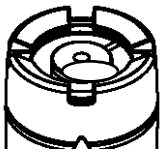
Twist a DO sensor
with holding a membrane cap tight.

6. **Check for membrane surface**

Check air bubbles in a membrane cap.



Good: Limited air bubbles



NG: Air bubbles of more than 5 mm in diameter

- NG → Replace a membrane cap again.
- Check that span calibration can be performed.

If the membrane cap is not attached correctly, sensitivity may be lost or response speed may decrease.

4.6 Troubleshooting

Note

If the sensor probe is removed while the control unit is indicating an error, errors cannot be canceled by using the ESC key. Either reconnect the sensor probe or restart the control unit.

4.6.1 Error displays

Error	Cause	Solution
Probe ADC error	Internal IC failure	Contact your nearest sales outlet to have the sensor probe repaired.
Probe EEPROM error/Factory	Internal IC failure	Turn the power OFF, then restart the system. If the error persists, initialize the system from the "System" menu. If the error still persists, contact your nearest sales outlet to have the sensor probe repaired.
Probe EEPROM error/User	Internal IC failure	Turn the power OFF, then restart the system. If the error persists, initialize the system from the "System" menu. If the error still persists, contact your nearest sales outlet to have the sensor probe repaired.
Turbidity sensor light source error	Turbidity sensor light source failure	Turn the power OFF, wipe off any water droplets on the probe, then remove the turbidity sensor. Check there are no water droplets around the turbidity sensor connector, then mount the sensor again. If the error persists, replace the turbidity sensor.
Turbidity sensor wiper motor error	The turbidity sensor wiper is not operating.	Press the ESC key. Check there are no obstacles near the wiper, then perform the measurement again. If the error persists, the motor will need to be replaced. Contact your nearest sales outlet to have the sensor probe repaired.
Probe capacitor error	Low battery voltage or internal IC failure	Turn the power OFF. Replace the display's batteries. If the error persists, contact your nearest sales outlet to have the sensor probe repaired.
Probe EEPROM error	Internal IC failure	Press the ESC key, then redo the operation. If the error persists, turn the power OFF, then restart the system (the current data will not be saved). If the error still persists, contact your nearest sales outlet to have the display repaired.
Probe board error	Probe board failure	Turn the power OFF. Contact your nearest sales outlet to have the sensor probe repaired.

Error	Cause	Solution
Zero-point calibration error	pH sensor 1. The pH standard solution is contaminated. 2. The pH-responsive membrane is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The pH-responsive membrane is torn.	pH sensor 1. Replace the standard solution with new solution. 2. Clean the pH-responsive membrane. 3. Refil the reference electrode's internal solution. 4. Replace the sensor.
	COND sensor 1. There is moisture on the sensor. 2. The sensor is dirty. 3. The COND sensor is broken.	COND sensor 1. Blow-dry the moisture off the sensor. 2. Clean the sensor. 3. Contact your nearest sales outlet.
	TURB sensor 1. There are air bubbles on the cell. 2. The cell window is dirty. 3. The sensor is being affected by ambient light. 4. The solution is dirty. 5. The TURB sensor has failed.	TURB sensor 1. Shake the sensor probe vigorously. 2. Clean the cell window. 3. Calibrate using the calibration cup provided. 4. Replace the solution with new solution. 5. Replace the TURB sensor.
	DO sensor 1. There are air bubbles in the internal solution. 2. The DO sensor has failed.	DO sensor 1. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 2. Replace the DO sensor.
	Water depth sensor 1. The water depth sensor is dirty. 2. The water depth sensor has failed.	Water depth sensor 1. Clean the water depth sensor. 2. Contact your nearest sales outlet.

Error	Cause	Solution
Span calibration error	pH sensor 1. The pH standard solution is contaminated. 2. The pH-responsive membrane is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The pH-responsive membrane is torn.	pH sensor 1. Replace the standard solution with new solution. 2. Clean the pH-responsive membrane. 3. Refil the reference electrode's internal solution. 4. Replace the sensor.
	ORP sensor 1. The ORP standard solution is contaminated. 2. The ORP electrode is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The ORP electrode has failed.	ORP sensor 1. Replace the standard solution with new solution. 2. Clean the ORP electrode. 3. Refil the reference electrode's internal solution. 4. Replace the ORP electrode.
	COND sensor 1. The calibration solution is not correct. 2. The sensor is dirty. 3. The COND sensor has failed.	COND sensor 1. Use the correct calibration solution for calibration. 2. Clean the sensor. 3. Contact your nearest sales outlet.
	TURB sensor 1. There are air bubbles on the cell. 2. The cell window is dirty. 3. The sensor is being affected by ambient light. 4. The solution is dirty. 5. The TURB sensor has failed.	TURB sensor 1. Shake the sensor probe vigorously. 2. Clean the cell window. 3. Calibrate using the calibration cup provided. 4. Replace the solution with new solution. 5. Replace the TURB sensor.
	DO sensor 1. The diaphragm is torn. 2. There are air bubbles in the internal solution. 3. The DO sensor has failed.	DO sensor 1. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 2. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 3. Replace the DO sensor.
	Temperature sensor The temperature sensor has failed.	Temperature sensor Contact your nearest sales outlet.
Calibration stability error	The calibration value of an individual parameter is not stable. 1. The sensor is dirty. 2. The sensor has not adjusted to the standard solution. 3. The temperature was unstable during calibration.	1. Clean the sensor. 2. Fill the transparent calibration cup with pH 4 standard solution, and wait for at least 20 minutes of conditioning before starting calibration. 3. Start calibration after the temperature has stabilized.
Turbidity calibration error	Error in turbidity measurement sequence	Turbidity calibration failed. Redo calibration after removing the displayed error.
Wet check	The cable connector is submerged.	Turn the power OFF and disconnect the cable connector. Wipe or blow-dry off all the water droplets on the probe. If the error persists, contact your nearest sales outlet to have the display and sensor probe repaired.
Power voltage error	The display's power board has failed.	This error could also be caused by poor cable contact. Turn the power OFF and disconnect the cable connector. Reconnect the connector and turn the power ON. If the error persists, contact your nearest sales outlet to have the display and sensor probe repaired.
Turbidity lamp power voltage error	The remaining battery level is low.	Turn the power OFF and replace the display's batteries with new ones.

Error	Cause	Solution
Display RTC error	The time display is incorrect.	Replace the coin battery.
Display FROM error	Internal IC failure	Contact your nearest sales outlet to have the control unit repaired.
Display EEPROM error	Internal IC failure	Contact your nearest sales outlet to have the control unit repaired.
Display save error	Insufficient memory space	Move data from the display, use the data operations screen to delete data, then redo the measurement.
Measurement sequence error	● When the measurement item is turbidity 1. The battery power is low. 2. The wiper is not operating normally. 3. The light source lamp is not lit. ● If items other than turbidity are also displayed 4. Board failure	1. Replace the batteries with new ones. 2. Check there are no obstacles near the wiper, then redo the measurement. If the error persists, the motor will need to be replaced. Contact your nearest sales outlet to have the sensor probe repaired. 3. Wipe off any water droplets on the probe, then remove the turbidity sensor. Check there are no water droplets around the turbidity sensor connector, then mount the sensor again. If the error persists, replace the turbidity sensor. 4. Contact your nearest sales outlet to have the sensor probe repaired.
Out of measurement range	The attempted measurement is outside the measurement range supported for that item.	The system must be used within its supported measurement ranges.
Last zero-point calibration invalid	pH sensor 1. The pH standard solution is contaminated. 2. The pH-responsive membrane is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The pH-responsive membrane is torn.	pH sensor 1. Replace the standard solution with new solution. 2. Clean the pH-responsive membrane. 3. Refil the reference electrode's internal solution. 4. Replace the sensor.
	COND sensor 1. There is moisture on the sensor. 2. The sensor is dirty. 3. The COND sensor has failed.	COND sensor 1. Blow-dry the moisture off the sensor. 2. Clean the sensor. 3. Contact your nearest sales outlet.
	TURB sensor 1. There are air bubbles on the cell. 2. The cell window is dirty. 3. The sensor is being affected by ambient light. 4. The solution is dirty. 5. The TURB sensor has failed.	TURB sensor 1. Shake the sensor probe vigorously. 2. Clean the cell window. 3. Calibrate using the calibration cup provided. 4. Replace the solution with new solution. 5. Replace the TURB sensor.
	DO sensor 1. There are air bubbles in the internal solution. 2. The DO sensor has failed.	DO sensor 1. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 2. Replace the DO sensor.
	Water depth sensor 1. The water depth sensor is dirty. 2. The water depth sensor has failed.	Water depth sensor 1. Clean the water depth sensor. 2. Contact your nearest sales outlet.
Out of measurement range	[See above.]	[See above.]
Last zero-point calibration invalid		

Error	Cause	Solution
Last span calibration invalid	pH sensor 1. The pH standard solution is contaminated. 2. The pH-responsive membrane is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The pH-responsive membrane is torn.	pH sensor 1. Replace the standard solution with new solution. 2. Clean the pH-responsive membrane. 3. Refil the reference electrode's internal solution. 4. Replace the sensor.
	ORP sensor 1. The ORP standard solution is contaminated. 2. The ORP electrode is dirty. 3. The concentration of the reference electrode's internal solution has changed. 4. The ORP sensor glass is broken.	ORP sensor 1. Replace the standard solution with new solution. 2. Clean the ORP electrode. 3. Refil the reference electrode's internal solution. 4. Replace the sensor.
	COND sensor 1. The calibration solution is not correct. 2. The sensor is dirty. 3. The COND sensor has failed.	COND sensor 1. Use the correct calibration solution for calibration. 2. Clean the sensor. 3. Contact your nearest sales outlet.
	TURB sensor 1. There are air bubbles on the cell. 2. The cell window is dirty. 3. The sensor is being affected by ambient light. 4. The solution is dirty. 5. The TURB sensor has failed.	TURB sensor 1. Shake the sensor probe vigorously. 2. Clean the cell window. 3. Calibrate using the calibration cup provided. 4. Replace the solution with new solution. 5. Replace the TURB sensor.
	DO sensor 1. The diaphragm is torn. 2. There are air bubbles in the internal solution. 3. The DO sensor has failed.	DO sensor 1. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 2. Replace the diaphragm with a new one, and fill the DO sensor with new internal solution. 3. Replace the DO sensor.
	Temperature sensor ● The temperature sensor has failed.	Temperature sensor ● Contact your nearest sales outlet.
Out of measurement range Last zero-point calibration invalid	[See above.]	[See above.]
Last span calibration invalid	The calibration value of an individual parameter is not stable. 1. The sensor is dirty. 2. The sensor has not adjusted to the standard solution. 3. The temperature was unstable during calibration.	1. Clean the sensors. 2. Fill the transparent calibration cup with pH 4 standard solution, and wait for at least 20 minutes of conditioning before starting calibration. 3. Start calibration after the temperature has stabilized.
Out of measurement range Last zero-point calibration invalid	[See above.]	[See above.]
Calibration value is factory default value.	Internal IC failure	Turn the power OFF, then restart the system. If the error persists, initialize the system from the "System" menu. If the error still persists, contact your nearest sales outlet to have the sensor probe repaired.

Error	Cause	Solution
Sample is unstable.	1. The concentration of the sample is unstable. 2. External light disturbance has affected the sensor. 3. Water has entered the turbidity sensor's connector.	1. Use a stirrer to agitate the sample during measurement. 2. Perform measurement away from direct sunlight. 3. Turn the power OFF, wipe off any water droplets on the probe, then remove the turbidity sensor. Check there are no water droplets around the turbidity sensor connector, then mount the sensor again. If the error persists, replace the turbidity sensor.

4.6.2 Error displays in sensor information

Error display	Cause	Solution
Measurement sequence error	Measurement sequence error	Turn the power OFF, then restart the system. If the error persists, have the probe repaired.
Out of measurement range	The measurement value is outside the measurement range.	Samples for measurement must be within the measurement range.
Last calibration invalid	The last calibration failed.	Redo calibration.
Calibration invalid	The calibration value is the factory default value.	Redo calibration.
Background unstable	The U-53 turbidity sensor is exposed to direct light.	Mount the guard cap and sensor guard and perform measurement away from direct sunlight.
	The turbidity value changed rapidly during measurement.	Measure a sample that has stable turbidity.

5 Specifications

Specification		Basic value	Model				
			U-51	U-52	U-52G	U-53	U-53G
Sensor probe	Measurement temperature range	−10°C to 55°C	✓	✓	✓	✓	✓
	Maximum sensor outer diameter	Approx. 96 mm					
	Sensor length	Approx. 340 mm					
	Cable length	2 m (standard) 10 m/30 m (options)					
	Mass	Approx. 1800 g					
	Auto calibration function	Uses pH 4 standard solution.					
	Measurement depth	30 m max.	✓	✓	✓	✓	✓
	Wet-part materials*3	PPS, glass, SUS316L, SUS304, FKM, PEEK, Q, titanium, FEP membrane, POM					
	Waterproofing standard	IP-68					
Control unit	Outer dimensions (W × D × H)	115 × 66 × 283 mm	✓	✓	—	✓	—
		115 × 66 × 335 mm	—	—	✓	—	✓
	Mass	Approx. 800 g	✓	✓	✓	✓	✓
	LCD	320 × 240 mm graphic LCD (monochrome) with backlight	✓	✓	✓	✓	✓
	Memory data items	10000	✓	✓	✓	✓	✓
	Communication interface	USB peripheral	✓	✓	✓	✓	✓
	Batteries	C-size dry cells (×4)	✓	✓	✓	✓	✓
	Waterproofing standard	IP-67	✓	✓	✓	✓	✓
	GPS unit	● Reception method (12 channel parallel) ● Measurement precision [With PDOP (high precision): 30 m or less (2 drms)]	—	—	✓	—	✓
	Estimated battery life*1	—	70 hours (no backlight)			500 measurements (no backlight)	
	Storage temperature range	−10°C to 60°C	✓	✓	✓	✓	✓
	Ambient temperature range	−5°C to 45°C					

Specification		Basic value	Model				
			U-51	U-52	U-52G	U-53	U-53G
pH measurement Two calibration	Measurement method	Glass electrode method	✓	✓	✓	✓	✓
	Range	pH 0 to 14					
	Resolution	0.01 pH					
	Precision*2	±0.1 pH					
Dissolved oxygen measurement ● Salinity conversion (0 to 70 PPT, automatic) ● Automatic temperature compensation	Measurement method	Polarographic method	✓	✓	✓	✓	✓
	Film thickness	25 μm					
	Range	0 mg/L to 50.0 mg/L					
	Resolution	0.01 mg/L					
	Precision*2	0 mg/L to 20 mg/L: ±0.2 mg/L 20 mg/L to 50 mg/L: ±0.5 mg/L					
Electrical conductivity measurement ● Auto range ● Automatic temperature conversion (25°C)	Measurement method	Four-AC-electrode method	✓	✓	✓	✓	✓
	Range	0 S/m to 10 S/m (0 mS/cm to 100 mS/cm)					
	Resolution	0.000 mS/cm to 0.999 mS/cm: 0.001 1.00 mS/cm to 9.99 mS/cm: 0.01 10.0 mS/cm to 99.9 mS/cm: 0.1 0.0 mS/m to 99.9 mS/m: 0.1 0.100 S/m to 0.999 S/m: 0.001 1.00 S/m to 9.99 S/m: 0.01					
	Precision*2	1% of full-scale (midpoint of two calibration points)					
Salinity measurement	Measurement method	Electrical conductivity conversion	✓	✓	✓	✓	✓
	Range	0 PPT to 70 PPT (parts per thousand)					
	Resolution	0.1 PPT					
	Precision	±3 PPT					
TDS (total dissolved solid) measurement ● Conversion coefficient setting	Measurement method	Electrical conductivity conversion	✓	✓	✓	✓	✓
	Range	0 g/L to 100 g/L					
	Resolution	0.1% of full-scale					
	Repeatability	±2 g/L					
	Precision	±5 g/L					
Seawater specific gravity measurement ● σt, σ0, σ15 display	Measurement method	Electrical conductivity conversion	✓	✓	✓	✓	✓
	Range	0 σt to 50 σt					
	Resolution	0.1 σt					
	Precision	±5 σt					

Specification		Basic value	Model				
			U-51	U-52	U-52G	U-53	U-53G
Temperature measurement	Measurement method	Platinum temperature sensor	✓	✓	✓	✓	✓
	Range	−10°C to 55°C					
	Resolution	0.01°C					
	Sensor	Platinum temperature sensor, JIS Class B (0.3 + 0.005 |t|)					
Turbidity measurement	Measurement method		–	LED forward 30° transmission/ scattering method		Tungsten lamp 90° transmission scattering method	
	Range			0 NTU to 800 NTU		0 NTU to 1000 NTU	
	Resolution			0.1 NTU		0.01 NTU	
	Precision*2			±5%of readout or ±1 NTU, whichever is larger		● ±0.5NTU (for 0 NTU to 10 NTU measurement range) ● 3% of readout or 1 NTU, whichever is larger (for 10 NTU to 1000 NTU measurement range)	
	Turbidity sensor wiper			–		✓	
Water depth measurement	Measurement method	Pressure method	–	–	✓	✓	✓
	Range	0 m to 30 m					
	Resolution	0.05 m					
	Precision*2	±0.3 m					
ORP (oxidation reduction potential) measurement	Measurement method	Platinum electrode method	✓	✓	✓	✓	✓
	Range	−2000 ～ +2000 mV					
	Resolution	1 mV					
	Precision*2	±15 mV					

*1: Battery life is estimated under following conditions.
- Continuous operation
- Using batteries: C-size alkaline dry cells
- Ambient temperature of the control unit: 20°C or more
- Backlight off

*2: The precision is defined by measuring the standard solution in the following cases.
- Turbidity and conductivity: after four point calibration
- pH and DO: after two point calibration
- Water depth and ORP: after one point calibration

*3: Metallic parts are made of stainless steel. Immersing in seawater may erode metallic parts.

6 Reference

6.1 Consumable parts

● Sensor

Name	Model	No.	Description
pH sensor	#7112	3014057312	Standard type pH sensor
pH sensor ToupH	#7113	3200170923	Tough glass type pH sensor
ORP sensor	#7313	3200170920	
DO sensor	#7543	3200170924	
Reference electrode	#7210	3200043582	
R bush unit	—	3200043587	Reference electrode liquid junction
TURB cell U-52	#7800	3200172803	For U-52/U-52G
TURB cell U-53	#7801	3200172800	For U-53/U-53G
Membrane cap	—	3200170194	For DO sensor

● Standard solution and inner solution

Name	Model	No.	Description
pH 4 (For automatic calibration) 500 mL	#100-4	3200043638	Standard solution for auto calibration. Also used for manual pH span calibration.
pH 4 (For automatic calibration) 4 L	#140-4	3200174430	
pH 7 500 mL	#100-7	3200043637	Standard solution for pH zero-point calibration.
pH 9 500 mL	#100-9	3200043636	Standard solution for pH manual span calibration.
Powder for ORP standard solution 10 packs	#160-51	3200043618	For ORP calibration.
Powder for ORP standard solution 10 packs	#160-22	3200043617	
Inner solution for DO sensor, 50 mL	#306	3200170938	Internal solution for DO sensor.
Internal solution for pH, 250 mL	#330	3200043641	Supplementary internal solution for pH reference electrode.

● Others

Name	Model	No.	Description
Silicone grease	—	3014017718	Silicone grease for coating sensor O-ring.
Sponge brush unit	—	3200169531	Brush for cleaning sensor probe.
O-ring set for reference electrode	—	3200169376	O-rings for reference electrode.
O-ring set for DO sensor	—	3200169426	O-rings for DO sensor.
Rubber cap set for sensor guard	—	3200169428	Rubber caps used between sensor guard and sensor probe.
O-ring set for pH and ORP sensor	—	3200169520	O-rings for pH and ORP sensors.
Wiper unit	—	3200169789	Rubber wiper for U-53/U-53G turbidity sensors.
Protective cap (blk) for pH sensor	—	3200175019	Cap attached to tip of pH sensor for sensor probe storage.
Rubber cap (whit) for DO sensor	—	3200175020	Cap attached to tip of DO sensor for sensor probe storage.

6.2 Options sold separately

Name	Model	No.	Description
Bag	U-5030	3200174772	Storage bag for sensor probes and flow cell. Can be carried in one hand.
Flow cell assy	—	3200156570	Used when collecting measurement samples by pump.
Probe guard	—	3200167002	Used for taking measurements in locations where there is a current or where there is a thick layer of sludge.
Communication cable	—	3200174823	A PC connection cable. Comes with data collection software.

6.3 pH measurement

6.3.1 Principle of pH measurement

U-50 series use the glass electrode method for pH measurements. The glass electrode method measures a potential difference between the glass film for pH and the reference electrode. For more information, refer to “JIS Z 8802 pH measurement method”.

6.3.2 Temperature compensation

The electromotive force generated by the glass electrode changes depending on the temperature of the solution.

Temperature compensation is used to compensate for the change in electromotive force caused by temperature.

This function does not compensate the change in pH caused by the temperature of the solution. When pH is to be measured, the temperature of the solution must be recorded along with that pH value, even if a pH meter has automatic temperature compensation function. If the solution temperature is not recorded, the results of the pH measurement may be meaningless.

6.3.3 Standard solutions

When measuring pH, the pH meter must be calibrated using standard solution. There are five kinds of standard solutions specified in “JIS Z 8802 pH measurement”. For normal measurement, two of standard solutions with pH of 4, 7, and 9 are sufficient to accurately calibrate the meter.

For standard solutions, refer to “JIS Z 8802 pH measurement”.

pH 4 standard solution: (Phthalate)	0.05 mol/L potassium hydrogen phthalate aqueous solution
pH 7 standard solutio: (Neutral phosphate)	0.025 mol/L potassium dihydrogenphosphate, 0.025 mol/L disodium hydrogenphosphate aqueous solution
pH 9 standard solution: (Borate)	0.01 mol/L sodium tetraborate aqueous solution

Table 2 pH values of pH standard solutions at various temperatures settings

Temp. (℃)	pH 4 standard solution Phthalate	pH 7 standard solution Neutral phosphate	pH 9 standard solution Borate
0	4.01	6.98	9.46
5	4.01	6.95	9.39
10	4.00	6.92	9.33
15	4.00	6.90	9.27
20	4.00	6.88	9.22
25	4.01	6.86	9.18
30	4.01	6.85	9.14
35	4.02	6.84	9.10
40	4.03	6.84	9.07
45	4.04	6.84	9.04

6.4 DO measurement

6.4.1 Principle of DO measurement

Dissolved oxygen (DO) refers to the amount of oxygen that is contained in water.
The concentration of dissolved oxygen is generally given as mg/L or as a percentage value (the dissolved oxygen saturation ratio).
Dissolved oxygen is essential for maintaining the self-purifying ability of rivers and seas and also for fish to live. The concentration of dissolved oxygen acts as an indicator of water quality. It is often measured when processing waste water and managing water quality. Fig. 1 provides an overview of the principles behind dissolved oxygen sensor measurement.

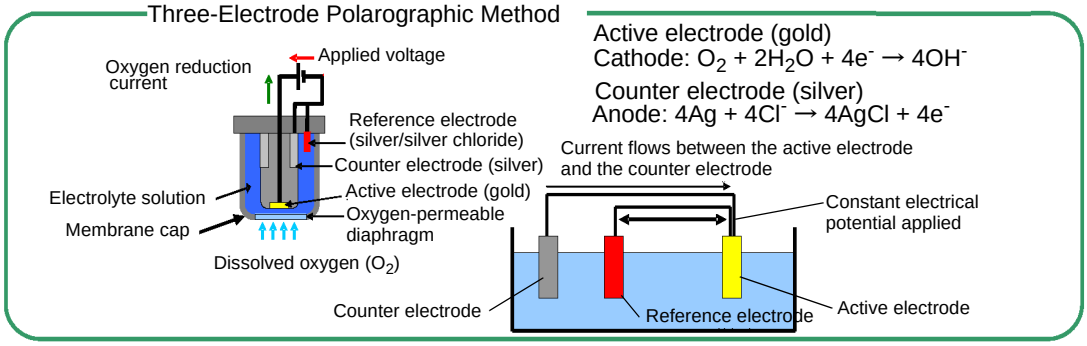


Fig. 1 Overview of principles behind dissolved oxygen sensor

The polarographic oxygen sensor is an enclosed sensor wherein voltage is applied to a cathode made of a precious metal (such as gold or platinum) and an anode also made of a precious metal (such as silver) via an external circuit, and a cap with an oxygen permeable diaphragm (membrane) is filled with electrolyte solution. As indicated in Fig. 1, the concentration of dissolved oxygen can be measured by measuring the current proportional to the amount of reduced oxygen when oxygen that has dispersed through the oxygen permeable diaphragm produces a reductive reaction on the surface of the active electrode (gold). The method of measuring dissolved oxygen based on the above principle is called the Membrane Electrode Method. Compared to the Chemical Analysis Method, which requires complicated pre-processing to alleviate the effect of reduced materials and oxidizing materials, this method allows dissolved oxygen to be measured very easily. It is also easy to remove undesired buildup from the silver electrode by polishing and cleaning if an insulator forms on it due to oxidation, making the method reusable.

6.4.2 Salinity calibration

When the solution and air come into contact and form an equilibrium (i.e. saturation), the relationship between the concentration of dissolved oxygen in the solution, C, [mol/L], and the partial pressure of oxygen in the air, Ps, [MPa/(mg/L)], can be represented by the following formula:

$$C = Ps/H$$

Where H [MPa/(mg/L)] is the Henry constant, a value that changes according to the composition of the solution. As H typically becomes larger as the salinity of the water increases, C becomes smaller.

The DO sensor detects the partial pressure of oxygen (Ps) in the above formula. Accordingly, if the DO sensor is immersed in deionized water saturated with air, or in an aqueous solution containing salt, the output current does not change, resulting in an erroneous measurement. For example, when salt is added to a sample, the amount of oxygen that can be dissolved in the solution decreases, but because the partial pressure of oxygen does not change, the value displayed by the control unit stays the same regardless of salt content. This concept is indicated in graph form below. (Fig. 2)

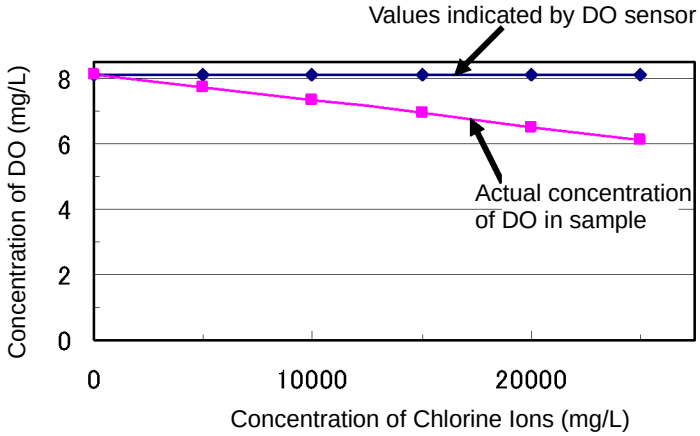


Fig. 2 Relationship between chlorine ion concentration and dissolved oxygen concentration

In samples with a high salt concentration, the solubility of oxygen is lower, but as the partial pressure of oxygen does not change, the value actually indicated on the control unit is higher than the actual value. In order to obtain a measurement of the concentration of dissolved oxygen in an aqueous solution that contains salt, it is therefore necessary to first perform salinity compensation. Conventionally, dissolved oxygen sensors have performed salinity compensation by inputting the salinity of the sample. This is fine as long as the salinity is already known. However, in most cases salinity is unknown, so even if dissolved oxygen sensors contained a salinity compensation function, it was of no practical use.

The U-50 Series can calculate and measure salinity in samples from electrical conductivity values, and can thus be used to automatically compensate for salinity.

6.5 Conductivity (COND) measurement

6.5.1 Four-AC-electrode method

Conductivity is an index of the flow of electrical current in a substance.

Salts dissolved in water are separated into cations and anions. Such solution is called electrolytic solution.

Electrolytic solution has the property of allowing the flow of current according to Ohm's law. This property is referred

to as ionic conductivity, since current flow is caused by ion movement in electrolytic solution.

Metals, on the other hand, allow the flow of current by means of electrons. This property is called electronic conductivity,

which is distinguished from ionic conductivity.

A cube with 1 m on each side, as shown in Fig. 3, is used to demonstrate an electrolytic solution. Two electrode plates are placed on opposite sides, and the cube is filled with solution. If the resistance between these two electrode plates is represented by r(Ω), the conductivity of the solution L($S \cdot m^{-1}$) is represented as L=1/r. S stands for Siemens, a unit of measurement of conductance.

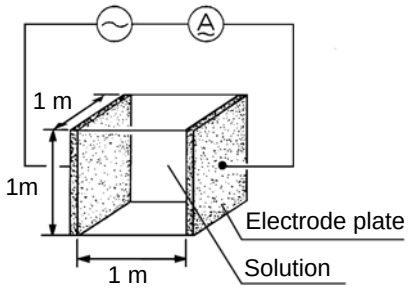


Fig. 3 Definition of conductivity

The most general method for measuring conductivity is based on the above principle, and is called the 2-electrode method.

In the 2-electrode method the influence of polarization cannot be ignored for solutions with high conductivity and conductivity cannot be measure accurately. In addition, contamination on the surface of the electrode increases apparent resistance, resulting in inaccurate measurement of conductivity.

The U-50 series has adopted the 4-electrode method to overcome these disadvantages of the the 2-electrode method.

As shown in Fig. 4, the U-50 series uses two voltage-detecting electrodes and two voltage-applying electrodes, for a total of four electrodes. The voltage-detecting electrodes are for detecting AC voltage, and the voltage-applying electrodes are for applying AC voltage.

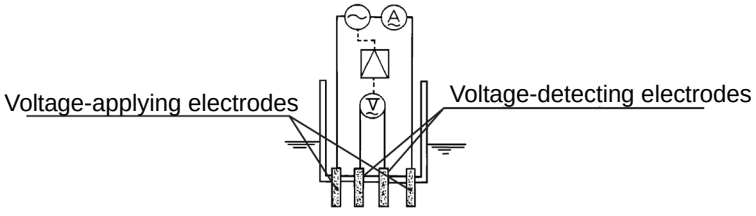


Fig. 4 Principle of the 4-electrode method

Let us assume that the current, I(A), flows in a sample of conductivity L – under automatic control of the voltage-applying electrodes – so that the voltage at the voltage-detecting-electrodes, E(V), remains constant at all times.

Then, the resistance of the sample, R(Ω), across the voltage-detecting electrodes is represented as R=E/I. The resistance, R, of the sample is inversely proportional to its conductivity, L. Accordingly, a measurement of current, Is,

of a standard solution of known conductivity, Ls, enables calculation of conductivity of a sample according to the formula L = Ls (I/Is) from the ratio L : Ls = I : Is.

Even in the 4-electrode method, polarization occurs, since AC current flows in the voltage-applying electrodes. The voltage-detecting electrodes are, however, free from the effects of polarization, since they are separated from the voltage-applying electrodes, and furthermore, current flow is negligible. Therefore, the 4-electrode method is an excellent method to enable measurement of conductivity covering a very high range.

6.5.2 SI units

New measurement units, called SI units, have been in use from 1996. Accordingly, the U-50 series also uses SI units. The following conversion table is provided for people who use the conventional kind of conductivity meter.

Note that along with the change in unit systems, the measurement values and cell counts have also changed.

	Former units	→	SI unit
Measurement value	0.1 mS/cm	→	0.01 S/m
	1 mS/cm	→	0.1 S/m
	100 mS/cm	→	10 S/m

6.5.3 Temperature coefficient

In general, the conductivity of a solution varies largely with its temperature.

The conductivity of a solution depends on the ionic conductivity, described earlier. As the temperature rises, conductivity becomes higher since the movement of the ions becomes more active.

The temperature coefficient shows the change in % of conductivity per °C, with a certain temperature taken as the reference temperature. This is expressed in units of %/°C. The temperature coefficient assumes the premise that the conductivity of a sample changes linearly according to temperature.

Strictly speaking, with actual samples, however, conductivity changes along a curve. Furthermore, the cuve varies with the type of sample. In the ranges of smaller temperature changes, however, samples are said to have the temperature coefficient of 2%/°C (at reference temperature 25°C); this holds for most samples, except in certain special cases.

(The temperature coefficients for various types of solutions are listed on the next page.)

The U-50 series uses an automatic temperature conversion function to calculate conductivity at 25°C at a temperature

coefficient of 2 %/°C based on the measured value of the temperature. Results are displayed on the readout.

The U-50 series's temperature conversion function is based on the following formula.

$L_{25} = L_t / \{ 1 + K (t - 25) \}$

L_{25} : Conductivity of solution converted to 25°C

t : Temperature of solution at time of measurement (°C)

L_t : Conductivity of solution at t (°C)

K : Temperature coefficient (%/°C)

● Conductivity and temperature coefficient for various solutions

Conductivity and related temperature coefficients of representative substances (at 25°C) are shown in the table below.

Substance	Temp. (ºC)	Conc. (wt%)	Cond. (S/m)	Temp.coef. (%/°C)	Substance	Temp. (ºC)	Conc. (wt%)	Cond. (S/m)	Temp.coef. (%/°C)
NaOH	15	5	19.69	2.01	NaCl	18	5	6.72	2.17
		10	31.24	2.17			10	12.11	2.14
		15	34.63	2.49			15	16.42	2.12
		20	32.70	2.99			20	19.57	2.16
		30	20.22	4.50			25	21.35	2.27
		40	11.64	6.48	Na_2SO_4	18	5	4.09	2.36
KOH	15	25.2	54.03	2.09			10	6.87	2.49
		29.4	54.34	2.21			15	8.86	2.56
		33.6	52.21	2.36	Na_2CO_3	18	5	4.56	2.52
		42	42.12	2.83			10	7.05	2.71
NH_3	15	0.1	0.0251	2.46			15	8.36	2.94
		1.6	0.0867	2.38	KCl	18	5	6.90	2.01
		4.01	0.1095	2.50			10	13.59	1.88
		8.03	0.1038	2.62			15	20.20	1.79
		16.15	0.0632	3.01			20	26.77	1.68
HF	18	1.5	1.98	7.20			21	28.10	1.66
		4.8	5.93	6.66	KBr	15	5	4.65	2.06
		24.5	28.32	5.83			10	9.28	1.94
HCl	18	5	39.48	1.58			20	19.07	1.77
		10	63.02	1.56	KCN	15	3.25	5.07	2.07
		20	76.15	1.54			6.5	10.26	1.93
		30	66.20	1.52			—	—	—
H_2S0_4	18	5	20.85	1.21	NH_4Cl	18	5	9.18	1.98
		10	39.15	1.28			10	17.76	1.86
		20	65.27	1.45			15	25.86	1.71
		40	68.00	1.78			20	33.65	1.61
		50	54.05	1.93			25	40.25	1.54
		60	37.26	2.13	NH_4NO_3	15	5	5.90	2.03
		80	11.05	3.49			10	11.17	1.94
		100.14	1.87	0.30			30	28.41	1.68
		—	—	—			50	36.22	1.56
HNO_3	18	6.2	31.23	1.47	$CuSO_4$	18	2.5	10.90	2.13
		12.4	54.18	1.42			5	18.90	2.16
		31	78.19	1.39			10	32.00	2.18
		49.6	63.41	1.57			15	42.10	2.31
		62	49.64	1.57	CH_3COOH	18	10	15.26	1.69
H_3PO_4	15	10	5.66	1.04			15	16.19	1.74
		20	11.29	1.14			20	16.05	1.79
		40	20.70	1.50			30	14.01	1.86
		45	20.87	1.61			40	10.81	1.96
		50	20.73	1.74			60	4.56	2.06

6.6 Salinity (SAL) conversion

The U-50 series is designed to calculate salinity as well as the other parameters.

Note that the “salinity” here is the salinity of sea water. There is a constant relation between conductivity and salinity at certain temperatures.

Therefore, if data on the conductivity and temperature are available, the corresponding salinity can be known. In other words, the salinity measurement of the U-50 series is based on the principle of calculating the salt content, making use of the measured values of conductivity and temperature.

Note therefore, that measured results of all substances whose conductivity is detected are displayed as salinity. For example, the measured result is displayed as NaCl concentration, even if in fact the sample component is, hydrochloric acid (HCl).

6.7 TDS conversion

TDS is short for Total Dissolved Solids and means the total dissolved solid amount.

The conductivity of a solution is affected by the amount of salinity, minerals, and dissolved gases. That is, conductivity is an index that shows the total amount of all substances in the solution. Of these substances, TDS indicates only the amount of dissolved solids.

TDS can be used for a comparison of the state of substances composed of a single component such as NaCl. However, the use of TDS for the comparison of solutions of different types causes serious errors.

Conductivity and TDS are expressed by the following formulas.

Conductivity in SI units (S/m) ..... TDS(g/L) = L (S/m) $\times$ K $\times$ 10

TDS(g/L) = L (mS/m) $\times$ K $\div$ 100

Conductivity in the old units (mS/cm) ..... TDS(g/L) = L (mS/cm)$\times$ K

K = TDS coefficient

Initial settings use the values listed in the table (Page 80) that generally uses TDS coefficients.

For accurate TDS comparisons, find the TDS coefficient from measured conductivity values. Then set the value thus obtained and make measurements.

6.8 σt conversion

- **Specific gravity of seawater**

The density and specific gravity of seawater are equal numerically and generally are not distinguished strictly. Since seawater density ρ is between 1.000 and 1.031, 1 is subtracted from ρ and σ is obtained by multiplying the value by 1000.

The resultant value is used as the specific gravity of seawater.

$\sigma = (\rho - 1) \times 1000$

The density of seawater ρ is expressed by function of temperature, hydraulic pressure, and salinity. The density of seawater under the atmospheric pressure is expressed as σ_t. The density of seawater under the atmospheric pressure is determined by temperature and salinity.

The U-50 Series models make salinity measurement through temperature measurements and conductivity conversion and find σ_t through calculations.

In Japan σ_{15} at 15°C is called a standard specific gravity and widely used while in foreign countries σ_0 at 0°C is employed. σ_{15} and σ_0 are determined by the function of salinity.

In ocean surveys, in particular, these values σ_t, σ_{15}, and σ_0 are more widely used than conductivity and salinity and, in the U-50 Series models, newly added as measurement components.

6.9 Turbidity (TURB) measurement

6.9.1 Principle of turbidity measurement

U-52 and U-53 sensors measure turbidity using the Transmitting and Scattering Method shown in Fig. 5. U-52 sensors use a pulse light LED (infra-red emitting diode) as a light source, and detect scattered light from a 30° angle off center. U-53 sensors use a tungsten lamp as a light source and detect scattered light from a 90° angle. Both models display turbidity as a ratio of scattered light to transmitted light to reduce the affect of the color of the sample. The U-53 method conforms to EPA Method 180.1, and employs wipers to reduce the affect of air bubbles.

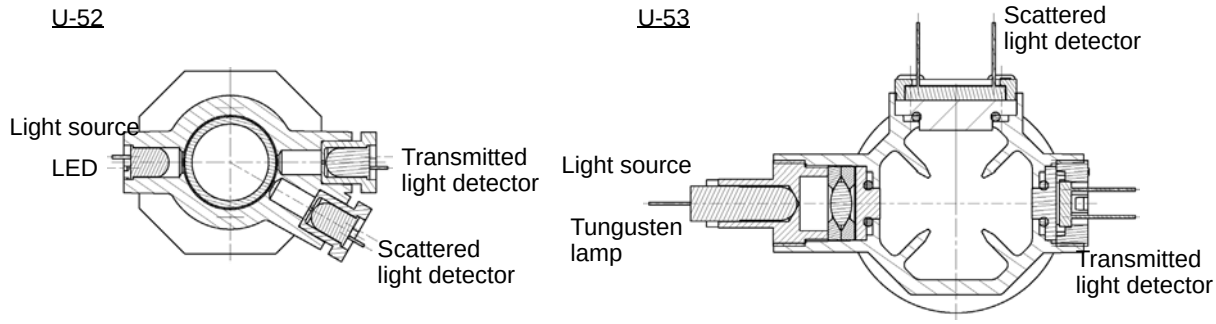


Fig. 5 Turbidity cell

6.9.2 Standard solution

U-50 series can perform calibration using formazin (NTU) or kaolin standard solutions as a turbidity standard solution. However, units for the solution used for calibration should be displayed in measurements. Do not use more than 400 mg/L of kaolin standard solution because it increases precipitation speed, resulting in measurement error.

6.10 Depth (DEPTH) measurement

6.10.1 Principle of depth measurement

For the W-22XD and W-23XD models, depth measurement can be made through use of a pressure gauge. The principle of the depth measurement uses the relation between depth and pressure.

Although the measurement with the depth sensor is affected by atmospheric pressure, the depth sensor, however, makes zero-point adjustments through the automatic calibration before measurements.

6.10.2 Influence of temperature and calibration

The depth sensor depends greatly on temperature. For a wide difference between the temperature at which the sensor has been automatically calibrated and the temperature of the measurement sample, the sensor can make depth measurements with a higher accuracy by the following method:

1. **Immerse the depth sensor of the sensor probe in the sample.**
2. **Keep the sensor immersed in the sample for about 30 minutes until the temperatures of the sensor and the sample are the same.**
3. **Then make the zero calibration of the sensor manually.**

6.11 Oxidation reduction potential (ORP) measurement

6.11.1 Principle of ORP measurement

ORP is an abbreviation for oxidation-reduction potential. ORP is the energy level (potential) determined according to the state of equilibrium between the oxidants (M^{Z+}) and reductants $M^{(Z-N)+}$ that coexist within a solution.

$$M^{Z+} + ne^- \Leftrightarrow M^{(Z-N)+} \quad \cdots (1)$$

If only the solution, forming the ORP measuring system shown in Fig. 6. The difference of potential between two electrodes is generally expressed by the following equation.

$$E = E_0 - \frac{RT}{nF} \ln \frac{a_M{}^{(z-n)+}}{a_M{}^{z+}} \quad \cdots (2)$$

E: Electric potential E_0 : Constant R: Gas constant T : Absolute temperature
n: Electron count F : Faraday constant a : Activity

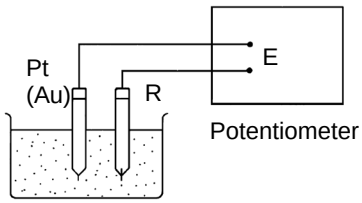


Fig. 6 Measuring mV

For example, for a solution in which trivalent iron ions coexist with bivalent iron ions, equations 1 and 2 would be as follows.

$$Fe^{3+} + e^- \Leftrightarrow Fe^{2+} \quad \cdots (1)$$

$$E = E_0 - \frac{RT}{F} \ln \frac{a_{Fe}{}^{2+}}{a_{Fe}{}^{3+}} \quad \cdots (2)$$

When only one type of state of equilibrium uniquely by equation (Fe^{3+}) and the reductant (Fe^{2+}) (using the equation $a_{Fe}{}^{2+}/ a_{Fe}{}^{3+}$). Actually, however many kinds of states of equilibrium exist simultaneously between various kinds of ions, in most solutions. This means that under actual circumstances, ORP cannot be expressed using the simple equation shown above and that the physical and chemical significance with respect to the solution is not very clear.
In this respect, the value of ORP must be understood to be only one indicator of the property of a solution. The measurement of ORP is widely used, however, as an important index in the analysis of solutions (potentiometric titration) and in the waste water treatment.

6.11.2 Standard electrode (reference electrode) types and ORP

The ORP is obtained comparing with corresponding reference electrode employed.
If different kinds of reference electrodes are used for measurement, the ORP value of the same solution may appear to be different. HORIBA's reference electrode uses Ag/AgCl with 3.33 mol/L KCl as inner solution. According to general technical literature, normal hydrogen electrodes (N.H.E.) are often used as the standard electrode.
The relationship between N.H.E. and the ORP that is measured using an Ag/AgCl with 3.33 mol/L KCl electrode is expressed by the following equation.
$E_{N.H.E.} = E + 206 - 0.7(t - 25)$ mV t = 0 - 60°C
$E_{N.H.E.}$: Measured ORP value using N.H.E. as the reference electrode

E: Measured ORP value using Ag/AgCl with 3.33 mol/L KCl as the reference electrode
Potential sign
Standard ORP is expressed in the following way, in literature related to electrochemistry and analytical chemistry.

A $Li^{+} + e^{-} \rightarrow Li$
$E_0 = -3.024$ V VS N.H.E.

However, in some literature, the "+" and "-" signs are reversed.

B $Li \rightarrow Li^{+} + e^{-}$
$E_0 = +3.024$ V VS N.H.E.

In expressions like B, above, the reaction is just reversed and there is no essential difference. But this kind of expression does invite confusion. The majority of the world, today, is consistent in its use of the signs as they are used in A, above.
For this reason, HORIBA, too, uses signs concerning ORP that are consistent with A, above.

HORIBA, Ltd.
2 Miyanohigashi, Kisshoin Minami-ku, Kyoto 601-8510 Japan
http://www.horiba.com

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For Sales & Service Contact
geotech
2650 E. 40th Ave. • Denver, CO 80205
Phone 303-320-4764 • Fax 303-322-7242
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LaMotte

2020we/wi TURBIDIMETER

1970-EPA
1970-ISO

GENERAL INFORMATION


2020we/wi QUICK START GUIDE

- Read the manual before using this meter.
- For the most accurate results, follow calibration and cleaning procedures before testing.
- To change settings, refer to the Options and Set Up section in the manual.
- Perform a turbidity calibration daily.

Factory Settings

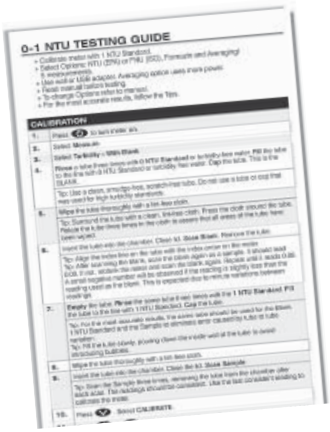
Averaging	Disabled
Turbidity Units	NTU
Turbidity Calibration	Formazin
Date Format	MM/DD/YYY
Power Save	5 minutes
Backlight	10 seconds
Language	English

Calibration

Perform a calibration over the smallest range possible. Use a standard that will bracket the sample range. Calibrate meter daily.

1. Press ON to turn meter on.
2. Select Mode.en
3. Select Turbidity – With Blank.
4. Rinse tube. Fill with BLANK. Dry tube.
5. Read Blank.
6. Rinse tube. Fill with STANDARD. Dry tube.
7. Read Sample.
8. Press ▼ and select Calibrate.
9. Press ▼ ▲ to adjust display to match standard value.
10. Press ENTER to accept the calibration.
11. Press ENTER to set the calibration.

Refer to the Quick Start Guide for simplified Calibration and Analysis procedures.


0-1 NTU TESTING GUIDE

- Calibrate meter with 1 NTU Standard.
- Select Options: NTU (EPA) or FNU (ISO), Formazin and Averaging.
- Use wall or USB adapter. Averaging option uses more power.
- Read manual before testing.
- To change Options refer to manual.
- For the most accurate results, follow the Tips.

CALIBRATION

1. Press ON to turn meter on.
2. Select Mode.en
3. Select Turbidity – With Blank.
4. Rinse a tube three times with 0 NTU Standard or turbidity free water. Fill the tube to the fill line (~5 NTU Standard or turbidity free water. Cap the tube. This is the BLANK.

   Tip: Use a clean, smudge-free, scratch-free tube. Do not use a tube or cap that was used for high turbidity standards.
5. Wipe the tube thoroughly with a lint-free cloth.

   Tip: Surround the tube with a clean, lint-free cloth. Press the cloth around the tube. Rotate the tube three times in the cloth to ensure that all areas of the tube have been wiped.
6. Insert the tube into the chamber. Close lid. Read Blank. Remove the tube.

   Tip: Align the index line on the tube with the index corner on the meter.

   Tip: After reading the blank, wipe the blank again as a sample. It should read 0.00. If not, restore the reading and scan the blank again. Repeat until it reads 0.00. A small negative number will be observed if the reading is slightly less than the reading used as the blank. This is expected due to minor variations between readings used as the blank.
7. Empty the tube. Rinse the screw tube 3 times with the 1 NTU Standard. Fill the tube to the line with 1 NTU Standard. Cap the tube.

   Tip: For the most accurate results, the same tube should be used for the Blank, 1 NTU Standard and the Sample to eliminate any CALIBRATION ERROR tube variations.

   Tip: Fill the tube slowly, pouring down the inside wall of the tube to avoid introducing bubbles.
8. Wipe the tube thoroughly with a lint-free cloth.
9. Insert the tube into the chamber. Close the lid. Read Sample.

   Tip: Scan the Sample three times, removing the tube from the chamber after each scan. The readings should be consistent. Use the last consistent reading to calibrate the meter.
10. Press ▼ Select CALIBRATE.

Refer to the Testing Guide for detailed Calibration and Analysis procedures for improving the accuracy of low range turbidity measurements.

INTRODUCTION

■ TURBIDITY

What is Turbidity?

Turbidity is an aggregate property of the solution, which is water in most cases. Turbidity is not specific to the types of particles in the water. The particles could be suspended or colloidal matter, and they can be inorganic, organic or biological. At high concentrations, turbidity is perceived as cloudiness, haze or an absence of clarity in the water. Turbidity is an optical property that results when light passing through a liquid sample is scattered. The scattering of light results in a change in the direction of the light passing through the liquid. This is most often caused when the light strikes particles in solution and is scattered backward, sideways and forward. If the turbidity is low, much of the light will continue in the original direction. Light scattered by the particles allows the particle to be ”seen” or detected in solution just as sunlight allows dust particles in the air to be seen.

In the past 10 years, turbidity has become more than just a measure of water clarity. Because of the emergence of pathogens such as Cryptosporidium and Giardia, turbidity now holds the key to assuring proper water filtration. In 1998, the EPA published the IESWTR (interim enhanced surface water treatment rule) mandating turbidities in combined filter effluent to read at or below 0.3 NTU. By doing so, the EPA hoped to achieve a 2 log (99%) removal of Cryptosporidium. There is presently consideration to lower this to 0.1 NTU. The trend has been to check the calibration of on-line turbidimeters with hand-held field units. The optical design and low detection limit of the 2020we/wi allows very accurate readings for such calibrations.

The meter also allows the user to choose the units of measure for expressing turbidity. While nephelometric turbidity unit (NTU) has been the standard for years, FNU (formazin nephelometric unit) and FAU (formazin attenuation unit) are now being used in ISO 7027 units. American Society of Brewing Chemists (ASBC) units and European Brewery Convention (EBC) units allow the brewing industry to check process waters.

How is Turbidity Measured?

Turbidity is measured by detecting and quantifying the scattering of light in water (solution). Turbidity can be measured in many ways. There are visual methods and instrumental methods. Visual methods are more suitable for samples with high turbidity. Instrumental methods can be used on samples with both high and low levels of turbidity.

Two visual methods are the Secchi Disk method and the Jackson Candle method. The Secchi Disk method is often used in natural waters. A black and white Secchi Disk is lowered into the water until it can no longer be seen. It is then raised until it can be seen again. The average of these two distances is known as the “Secchi Depth”. The Jackson Candle method uses a long glass tube over a standard candle. Water is added or removed from the tube until the candle flame becomes

indistinct. The depth of the water measured with a calibrated scale is reported as Jackson Turbidity Units (JTU). The lowest turbidity that can be determined with this method is about 25 NTU. There are two common methods for instruments to measure turbidity. Instruments can measure the attenuation of a light beam passing through a sample and they can measure the scattered light from a light beam passing through a sample. In the attenuation method, the intensity of a light beam passing through a turbid sample is compared with the intensity passing through a turbidity-free sample at 180° from the light source. This method is good for highly turbid samples. The most common instrument for measuring scattered light in a water sample is a nephelometer. A nephelometer measures light scattered at 90° to the light beam. Light scattered at other angles may also be measured, but the 90° angle defines a nephelometric measurement. The light source for nephelometric measurements can be one of two types to meet EPA or ISO specifications. The EPA specifies a tungsten lamp with a color temperature of 2,200–3,000 K. The units of measurement for the EPA method are nephelometric turbidity units (NTU). The ISO specifies a light emitting diode (LED) with a wavelength of 860 ± 30 nm and a spectral bandwidth less than or equal to 60 nm. The units of measurement for the ISO method are formazin nephelometric units (FNU). The 2020we meets the EPA specification and the 2020wi meets the ISO specification. The nephelometric method is most useful for low turbidity.

The 2020we/wi is a nephelometer that is capable of measuring turbidity by both the attenuation method and the nephelometric method. It uses a detector placed at 180° to the light source for high turbidity samples. It uses a detector placed at 90° to the light source for the nephelometric method for low turbidity samples. The 2020we/wi has a signal averaging option to improve the stability of readings on low turbidity samples.

The 2020we/wi has two different turbidity calibrations, formazin and Japan Standard. The formazin calibration is the EPA and ISO approved method of calibrating nephelometers. This calibration can be used with user prepared formazin standards or commercially purchased formazin standards. LaMotte Company approved AMCO™ standards labeled for use with the 2020we/wi can also be used with the formazin calibration. Stablcal® standards below 50 NTU should not be used to calibrate the 2020we/wi.

The Japan Standard calibration is a calibration for a Japanese Water Works standard. It is based on Japanese formulated polystyrene turbidity standards. This calibration should only be used to meet Japanese Water Works requirements. The Japanese polystyrene standards can only be purchased in Japan. Formazin, AMCO and Stablcal® standards cannot be used with this calibration.

Taking Turbidity Water Samples

Clean plastic or glass containers may be used for turbidity samples. Ideally, samples should be tested soon after collection and at the same temperature as when collected.

■ SAMPLE DILUTION TECHNIQUES

If a test result is out of the range of the meter, it must be diluted. The test should then be repeated on the diluted sample. The following table gives quick reference guidelines for dilutions of various proportions.

Amount of Sample	Deionized Water to Bring Final Volume to 10 mL	Multiplication Factor
10 mL	0 mL	1
5 mL	5 mL	2
2.5 mL	7.5 mL	4
1 mL	9 mL	10
0.5 mL	9.5 mL	20

All dilutions are based on a final volume of 10 mL, so several dilutions will require small volumes of the water sample. Graduated pipets should be used for all dilutions. If volumetric glassware is not available, dilutions can be made with the colorimeter tube. Fill the tube to the 10 mL line with the sample and then transfer it to another container. Add 10 mL volumes of deionized water to the container and mix. Transfer 10 mL of the diluted sample to the colorimeter tube and follow the test procedure. Repeat the dilution and testing procedures until the result falls within the range of the calibration. Multiply the test result by the dilution factor. For example, if 10 mL of the sample water is diluted with three 10 mL volumes of deionized water, the dilution factor is four. The test result of the diluted sample should be multiplied by four.

OPTIONS & SET UP

■ FACTORY DEFAULT SETTINGS

Settings that have user options have been set at the factory to default settings.

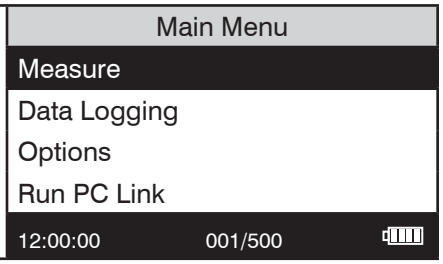
The factory default settings are:

Averaging	Disabled
Turbidity Units	NTU
Turbidity Calibration	Formazin
Date Format	MM-DD-YYYY
Power Save	5 minutes
Backlight	10 seconds
Language	English

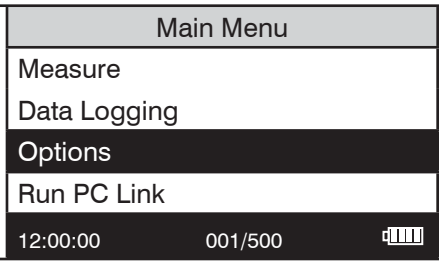
■ AVERAGING

The averaging option allows the user to average multiple readings. This option will improve the accuracy of samples with readings that may tend to drift with time. When the two, five or ten measurement option has been selected the final average is displayed. The averaging option is available only for turbidity. The default setting is disabled. To change the setting:

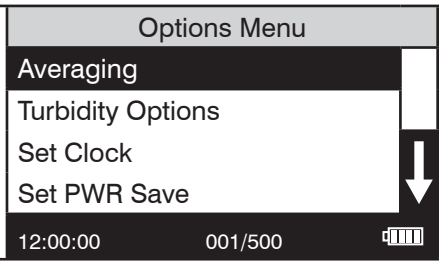
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.



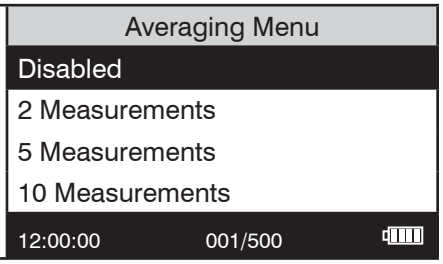
2. Press ▼ to scroll to **Options**.



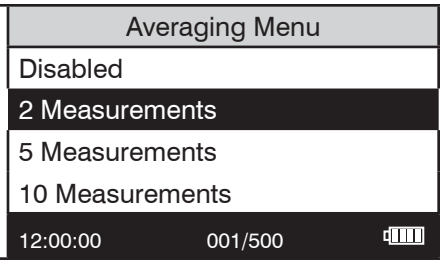
3. Press ENTER to select **Options**.



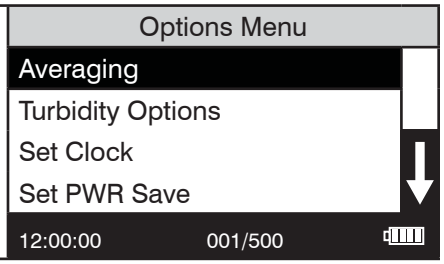
4. Press ENTER to select **Averaging**.



5. Press ▲ or ▼ to scroll to the desired option.



6. Press ENTER to save the selection. The screen will display **Storing...** for about 1 second and return to the **Options Menu**.



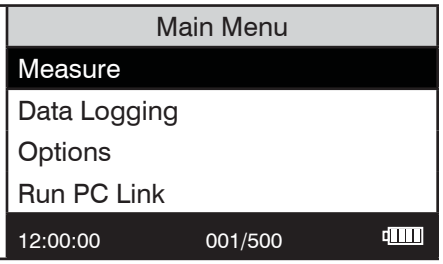
NOTE: When the **Averaging** option is enabled, more time will be required to display a reading and more power will be used.

■ TURBIDITY OPTIONS

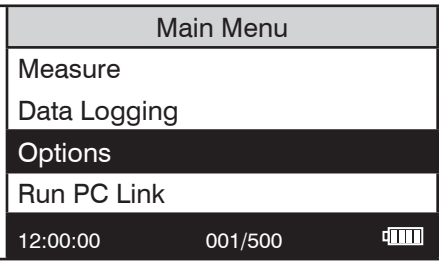
The default units are NTU and the default calibration curve is formazin. To change the settings:

Selecting Turbidity Units

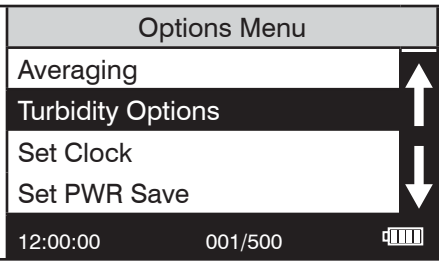
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.



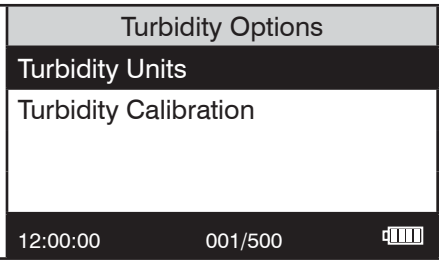
2. Press ▼ to scroll to **Options**.

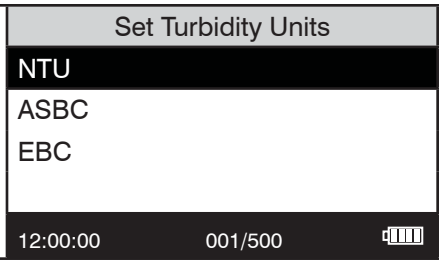


3. Press ENTER to select **Options**. Press ▼ to scroll to **Turbidity Options**.

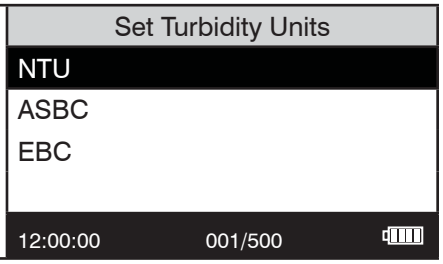


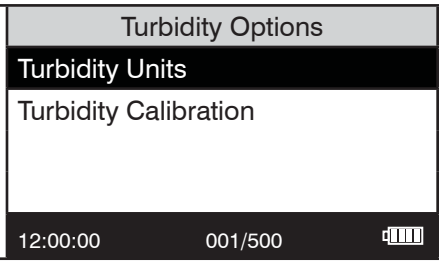
4. Press ENTER to select **Turbidity Options**.



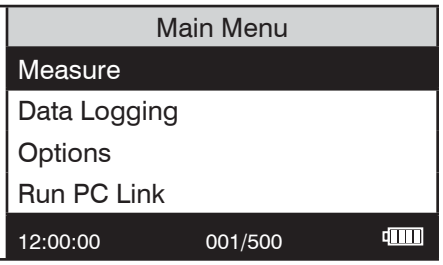
5. Press ENTER to select **Turbidity Units**.	

Available units are:
NTU (Nephelometric Turbidity Units)(2020we only)
FNU (Formazin Nephelometric Units) (2020wi only)
ASBC (American Society of Brewing Chemists)
EBC (European Brewery Convention)
NOTE: The meter will automatically switch to the attenuation mode above approximately 600 NTU or FNU. Measurements will be made with the 180° detector as indicated by AU or FAU on the display.

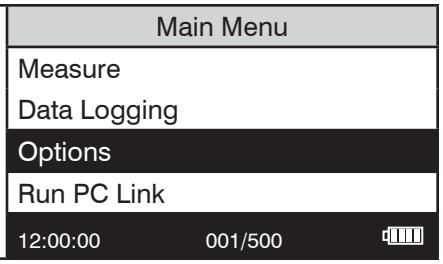
6. Press ^ or v to scroll to the desired units.	

7. Press ENTER to save the selection. The screen will display **Storing...** for about 1 second and return to the **Turbidity Options** menu. Press EXIT to return to a previous menu.	

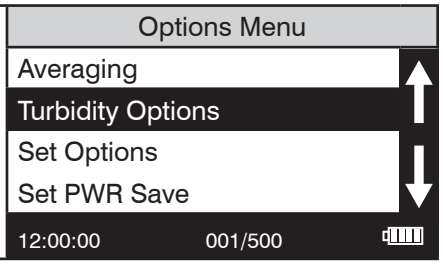
Selecting a Turbidity Calibration Curve

1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.	

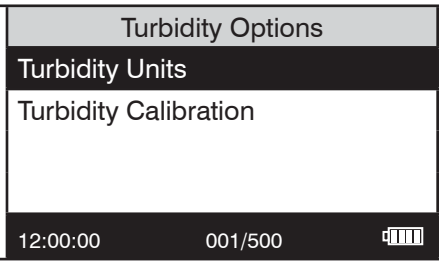
2. Press ▼ to scroll to **Options**.



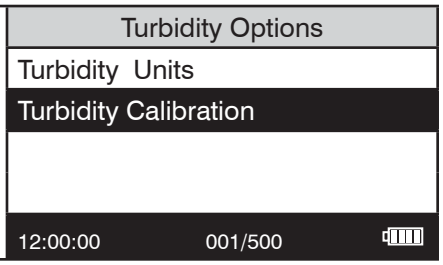
3. Press ENTER to select **Options**. Press ▼ to scroll to **Turbidity Options**.



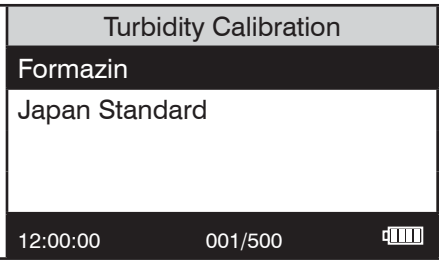
4. Press ENTER to select **Turbidity Options**.



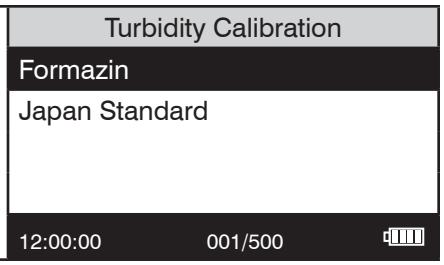
5. Press ▼ to scroll to **Turbidity Calibration**.



6. Press ENTER to select **Turbidity Calibration**.

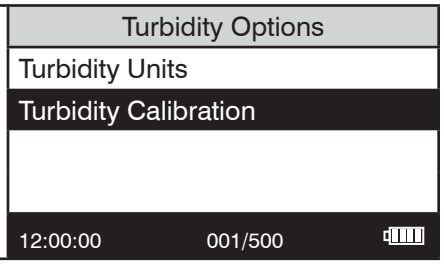


7. Scroll to the desired calibration option. Select a calibration option based on the composition of the standards that will be used to calibrate the meter.



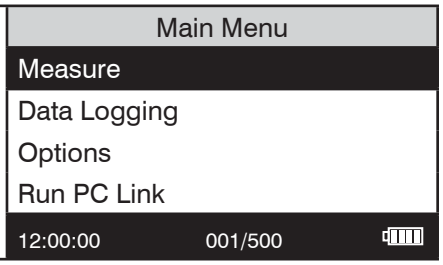
NOTE: Stablcal® standards below 50 NTU should not be used to calibrate the 2020we/wi. The diluent has a different refractive index than traditional formazin standards and will affect the results.

8. Press ENTER to save the selection. The screen will display **Storing...** for about 1 second and return to the **Turbidity Options** menu. Press EXIT to return to a previous menu.

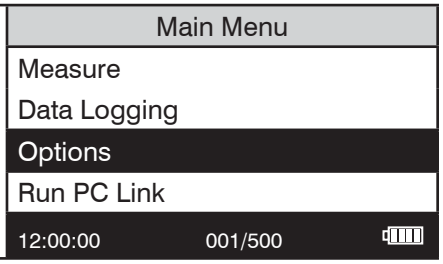


■ SETTING THE CLOCK

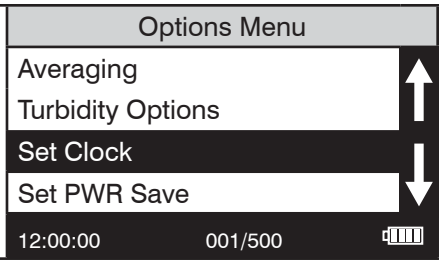
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.



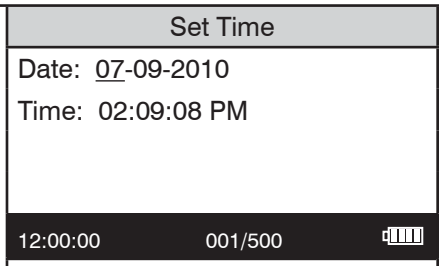
2. Press ▼ to scroll to **Options**.



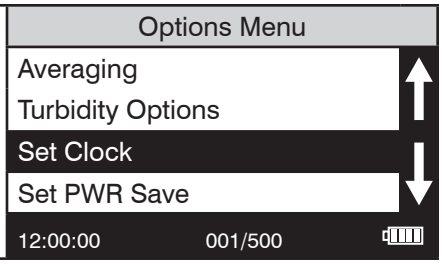
3. Press ENTER to select **Options**. Press ▼ to scroll to **Set Clock**.



4. Press ENTER to select **Set Clock**. The date is displayed as month-day-year. The time is displayed as hours:minutes:seconds AM/PM. Press ▲ or ▼ to the appropriate character and press ENTER to select. The cursor will move to the next character. Set all characters in the same manner. This is a scrolling menu.



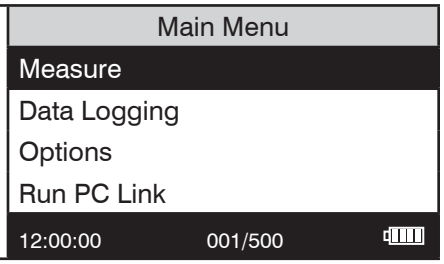
5. Press ENTER to select the final character. The time and date will be saved and the screen will return to the **Options Menu**.



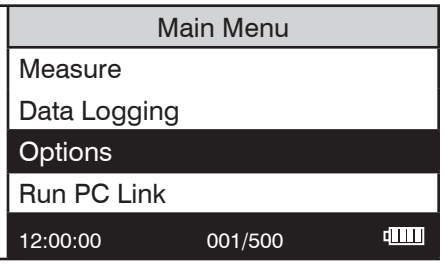
■ SETTING POWER SAVE

The power saving Auto Shutoff feature will turn the meter off when a button has not been pushed for a set amount of time. The default setting is disabled. To change the setting:

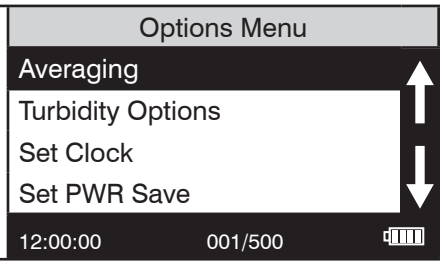
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.



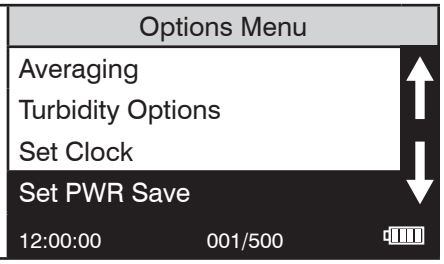
2. Press ▼ to scroll to **Options**.



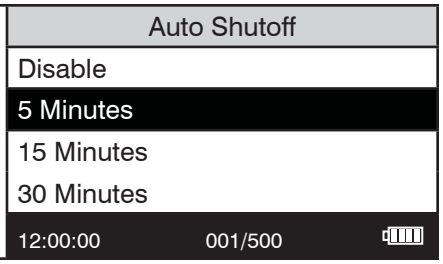
3. Press ENTER to select **Options**.



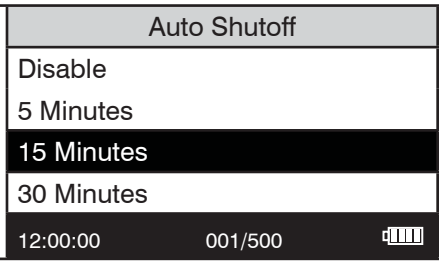
4. Press ▼ to scroll to **Set PWR Save.**



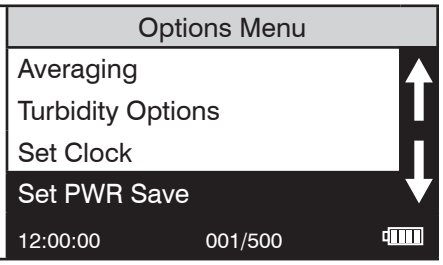
5. Press ENTER to select **PWR Save**.



6. Press ▼ ▼ to scroll to desired setting.



7. Press ENTER to save the selection. The screen will display **Storing...** for about 1 second and return to the **Options Menu**.

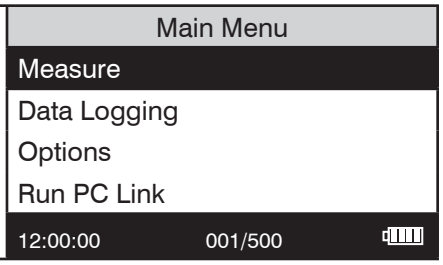


■ SETTING THE BACKLIGHT TIME

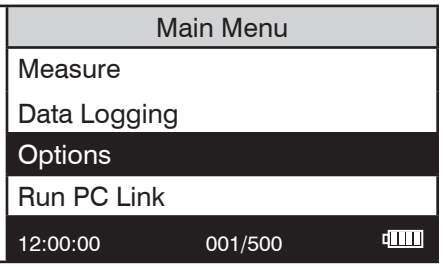
The backlight illuminates the display for enhanced viewing. If Button Control is chosen the backlight button on the key pad will act as an on/off switch and the backlight will remain on or off when the meter is being used. When one of the other settings – 10, 20 or 30 seconds – is chosen, the display will be illuminated for the specified amount of time after any button is pressed. As a precaution, the backlight will not illuminate during turbidity measurements to avoid interference from stray light.

NOTE: The backlight feature uses a significant amount of power. The longer the backlight is on, the more frequently the battery will have to be charged if the USB/Wall Charger is not being used.

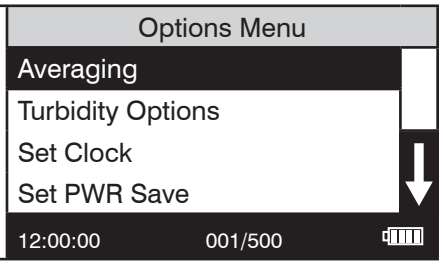
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.



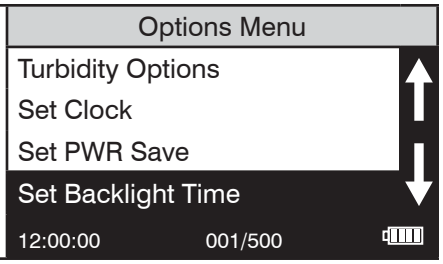
2. Press ⌄ to scroll to **Options**.



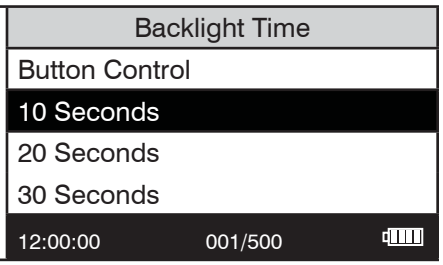
3. Press ENTER to select **Options**.



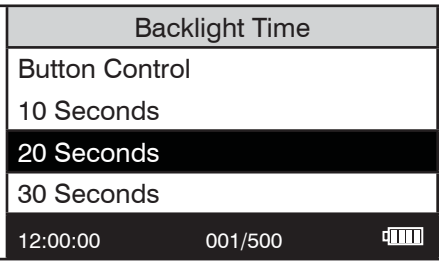
4. Press ▼ to scroll to **Set Backlight Time.**



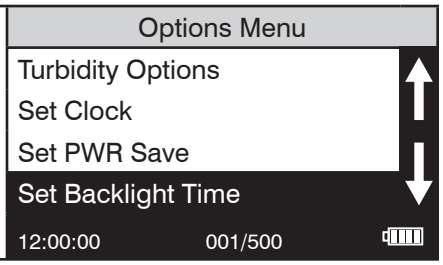
5. Press ENTER to select **Set Backlight Time.**



6. Press ▼ ▼ to scroll to desired setting.



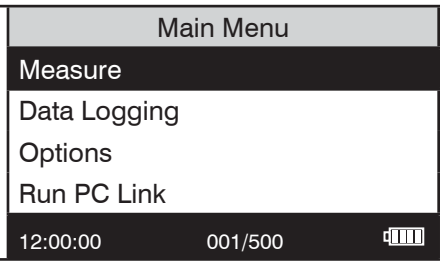
7. Press ENTER to save the selection. The screen will display **Storing...** for about 1 second and return to the **Options Menu**.



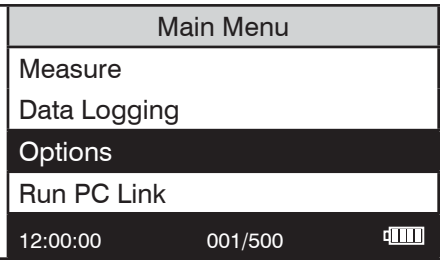
■ FACTORY RESET

Performing a Factory Reset will restore the factory default settings. All user-level calibrated settings will be lost.

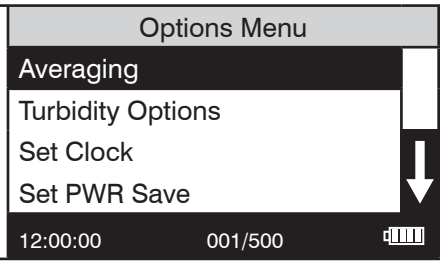
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.



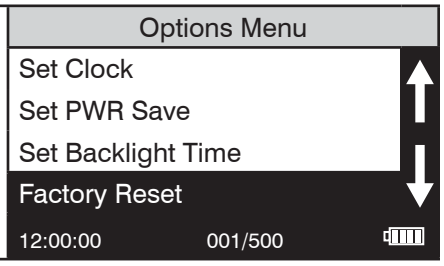
2. Press ▼ to scroll to **Options**.



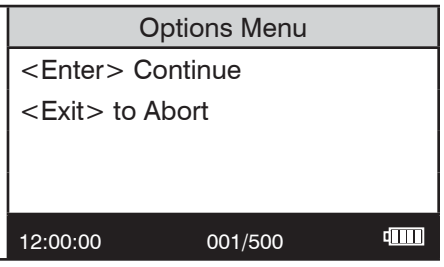
3. Press ENTER to select **Options**.



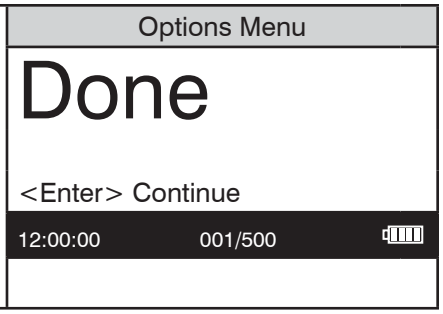
4. Press ▼ to scroll to **Factory Reset.**



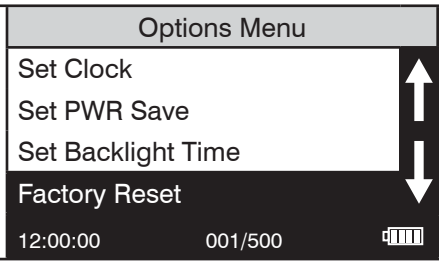
5. Press ENTER to select to **Factory Reset.**



6. Press ENTER to complete the Factory Reset. The screen will momentarily display **Writing**. The screen will display **Done** and return to the **Options Menu.** To retain the current user level calibration settings, press EXIT to abort the Factory Reset.



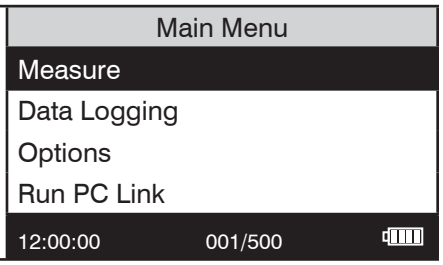
7. Press ENTER to return to the **Options Menu.**



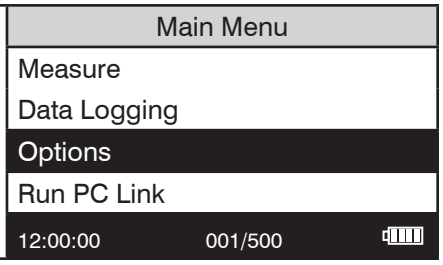
■ SELECTING A LANGUAGE

There are seven languages available in the 2020we/wi: English, Spanish, French, Portuguese, Italian, Chinese, and Japanese (Kana).

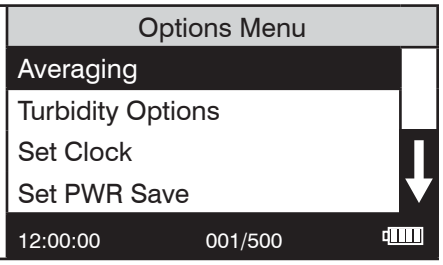
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.



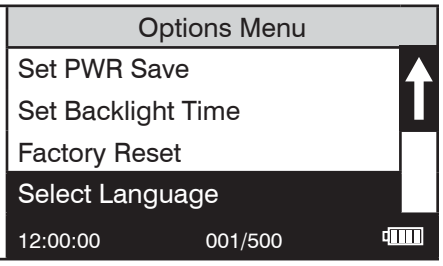
2. Press ▼ to scroll to **Options**.



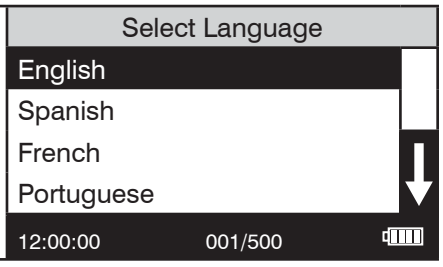
3. Press ENTER to select **Options**.



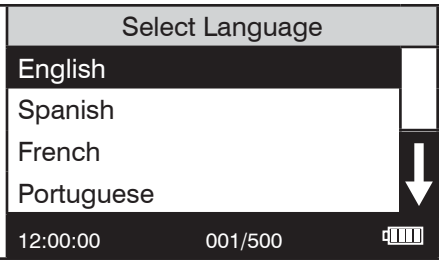
4. Press ▼ to scroll to **Select Language.**



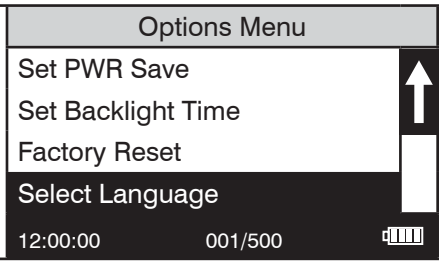
5. Press ENTER to select to **Select Language**.



6. Press ▲ or ▼ to scroll to desired language.



7. Press ENTER to select desired language. The screen will momentarily display, **Storing...**for about 1 second and return tot the **Options Menu**.



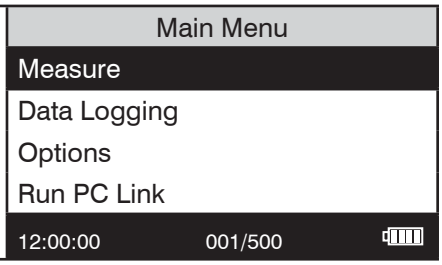
NOTE: If the meter unintentionally switches to another language, use the procedure above to reset the meter to the desired language. For example, to reset the meter to English:

1. Turn the meter on.
2. Press down arrow twice. Press ENTER.
3. Press down arrow seven times. Press ENTER.
4. Press ENTER.

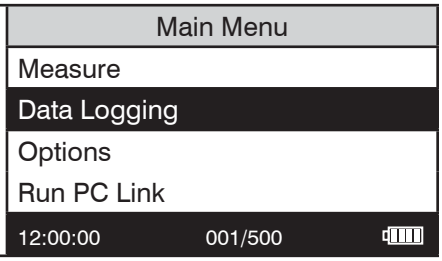
DATA LOGGING

The default setting for the data logger is enabled. The meter will log the last 500 data points. The counter in the center bottom of the display will show how many data points have been logged.

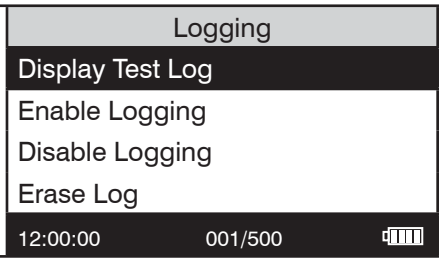
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.



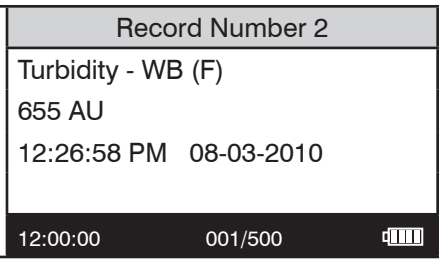
2. Press ▼ to scroll to **Data Logging**.



3. Press ENTER to select **Data Logging**.



4. Press ENTER to display the last data point and the time that it was logged.



5. Press ^ or v to scroll through the data points in the log.	Record Number 1 Turbidity - WB (F) 95.4 NTU 12:26:44 PM 08-03-2010 12:00:00 001/500

6. Press EXIT to return to the **Logging** menu. Press ^ or v to scroll to disable the logging options or erase the log. Press ENTER to select the option. The screen will display **Storing...** for about 1 second and return to the **Logging Menu**.	Logging Display Test Log Enable Logging Disable Logging Erase Log 12:00:00 001/500

CALIBRATION & ANALYSIS

■ CALIBRATION

Turbidity Standards

Only use AMCO or formazin standards with the 2020we/wi. StablCal® standards below 50 NTU should not be used to calibrate the 2020we/wi. The diluent used in the StablCal® standards has a different refractive index than traditional formazin standards and will affect the results. The concentration of the calibration standard should be similar to the expected concentration of sample that will be tested. The following standards are available from LaMotte Company:

1480 0 NTU Standard, 60 mL (EPA or ISO)

1450 1 NTU Standard, 60 mL (EPA)

1453 1 NTU Standard, 60 mL (ISO)

1451 10 NTU Standard, 60 mL (EPA)

1454 10 NTU Standard, 60 mL (ISO)

1452 100 NTU Standard, 60 mL (EPA)

1455 100 NTU Standard, 60 mL (ISO)

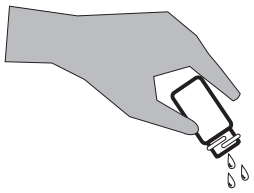
Turbidity Calibration Procedure

The default units are NTU and the default calibration curve is formazin as indicated by (F) in the Menu bar. For the most accurate results, a user calibration should be performed. The Japan Standard calibration mode, as indicated by (J) in the Menu bar, should be used only with Japanese Polystyrene Standards (0-100 NTU). To change the settings see the Set Up Instructions on page 9.

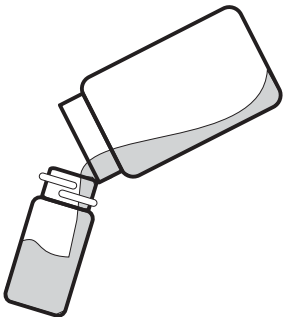
For the most accurate results, perform a calibration over the smallest range possible. Use a calibration standard that, along with the blank, brackets the range of samples that will be tested. For example, if the samples that are to be tested are expected to be below 1 NTU, more accurate results will be obtained by calibration with a blank and a 1 NTU standard as opposed to a blank and a 100 NTU standard.

It is recommended that the meter be calibrated daily.

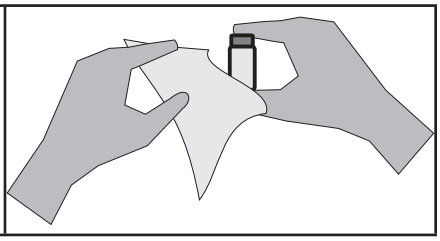
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.	Main Menu Measure Data Logging Options Run PC Link 12:00:00 001/500

2. Press ENTER to select **Measure**.	Measure Menu Turbidity - No Blank Turbidity - With Blank 12:00:00 001/500
3. Press ▼ to scroll to **Turbidity - With Blank**.	Measure Menu Turbidity - No Blank Turbidity - With Blank 12:00:00 001/500
4. Press ENTER to select **Turbidity - With Blank.**	Turbidity WB (F) Scan Blank Scan Sample 12:00:00 001/500
5. Rinse a clean tube (0290) three times with the blank. If samples are expected to read below 1 NTU the meter should be blanked with a 0 NTU Primary Standard or prepared turbidity-free (<0.1 NTU) water. For the most accurate results, use the same tube for the blank and the sample.	

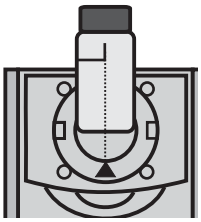
6. Fill the tube to the fill line with the blank. Pour the blank down the inside of the tube to avoid creating bubbles. Cap the tube.



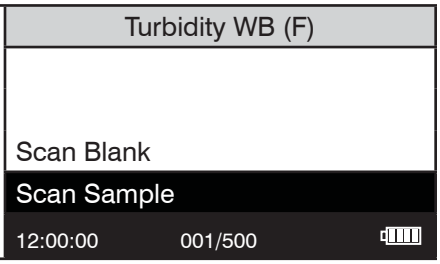
7. Wipe the tube thoroughly with a lint-free cloth.



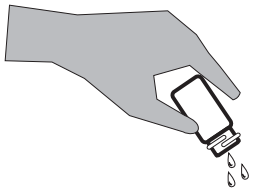
8. Open the meter lid. Insert the tube into the chamber. Align the index line on the tube with the index arrow on the meter. Close the lid.



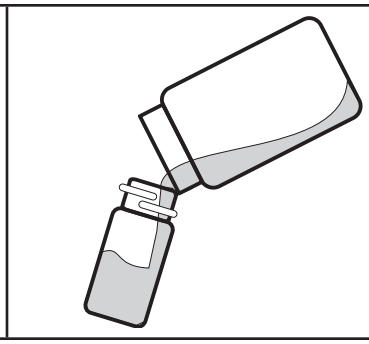
9. Press ENTER to scan the blank. The screen will display **Blank Done** for about 1 second and then return to the **Turbidity - With Blank Menu**.



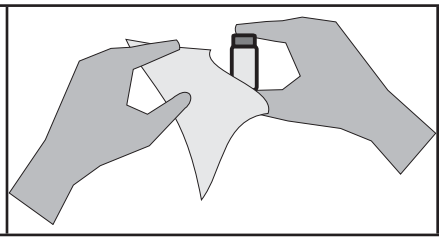
10. Rinse a clean tube (0290), or the same tube, three times with the standard.



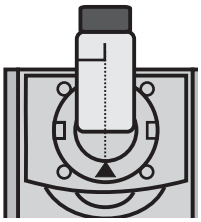
11. Fill the tube to the fill line with the standard. Pour the standard down the inside of the tube to avoid creating bubbles. Cap the tube.



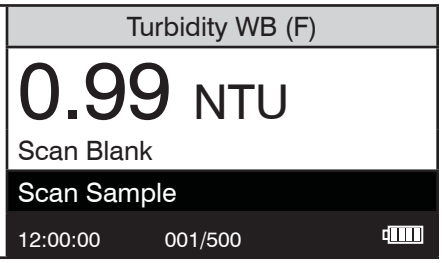
12. Wipe the tube thoroughly with a lint-free cloth.



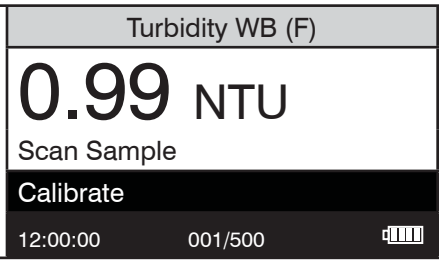
13. Open the meter lid. Insert the tube into the chamber. Align the index line on the tube with the index arrow on the meter. Close the lid.



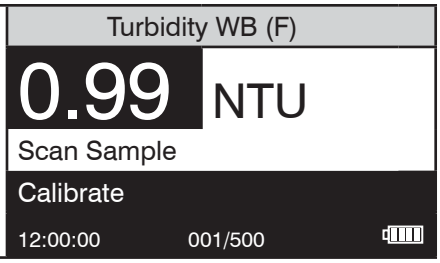
14. Press ENTER to scan the standard. The screen will display **Reading** for about 1 second. The result will appear on the screen.



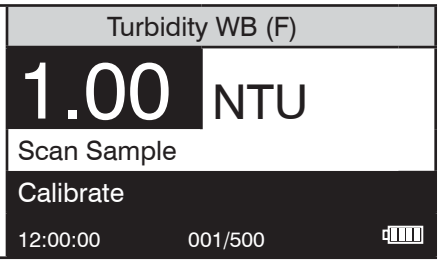
15. Press ▼ to scroll to **Calibrate**.



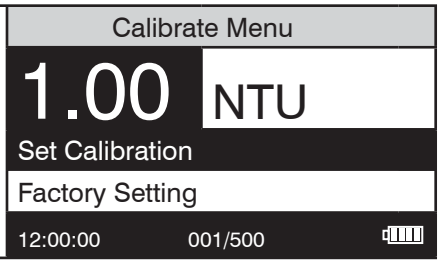
16. Press ENTER to select **Calibrate**. A reverse font (dark background with light characters) will appear to indicate that the reading can be adjusted.



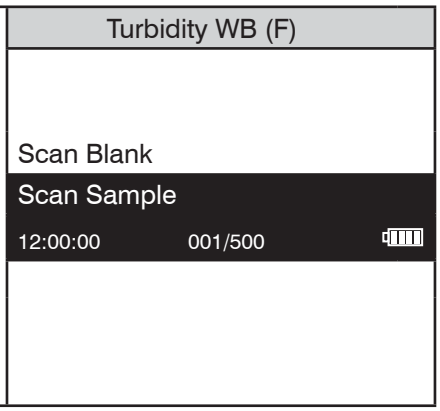
17. Press ^ or v to scroll to the concentration of the standard, 1.00 in the example. Note: The allowable adjustment is ±10%.



18. Press ENTER to select **Caibrate**. Two menu choices will be offered, **Set Calibration** and **Factory Setting**.



19. Press ENTER to select **Set Calibration** and save the calibration. Press ^ or v to scroll and select **Factory Setting** to revert to the factory calibration. The meter will momentarily display **Storing...** and return to the **Turbidity -Without Blank** menu. The calibration has now been saved and the meter can be used for testing.

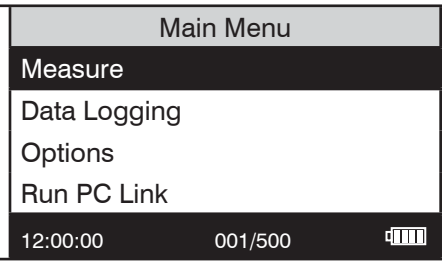


NOTE: For the greatest accuracy during the calibration procedure, be sure that after the meter is blanked and the blank is scanned as a sample, the reading is 0.00. If not, reblank the meter and scan the blank again until it reads 0.00. When scanning the calibration standards as the sample, scan the calibration standard three times removing the tube from the chamber after each scan. The readings should be consistent. Use the last consistent reading to calibrate the meter. If the readings are not consistent, avoid using an aberrant reading to calibrate the meter.

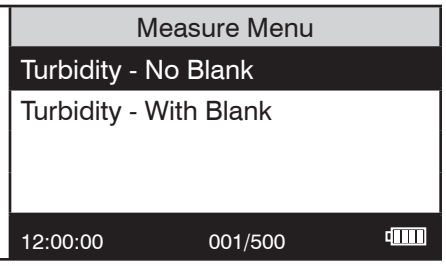
■ ANALYSIS WITHOUT BLANKING PROCEDURE

To obtain the most accurate results the meter should be blanked before measuring a sample. The blanking step is not as critical for samples above 10 NTU. The meter should always be blanked before reading samples below 10 NTU.

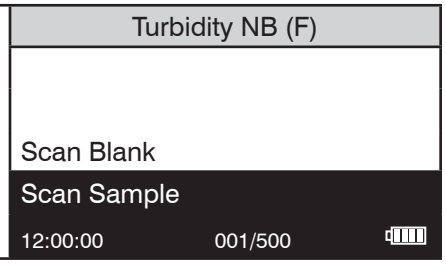
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.



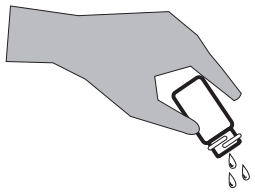
2. Press ENTER to select **Measure**.



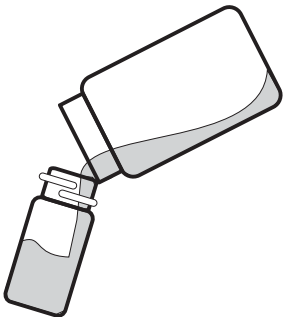
3. Press ENTER to select **Turbidity - No Blank**.



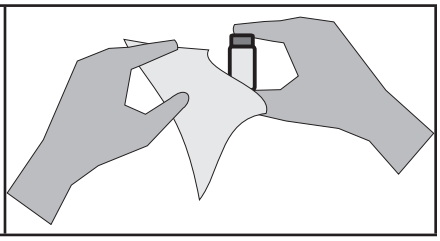
4. Rinse a clean tube (0290) three times with the sample.



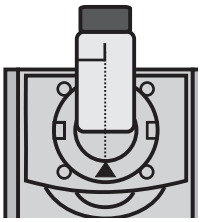
5. Fill the tube to the fill line with the sample. Pour the sample down the inside of the tube to avoid creating bubbles. Cap the tube.



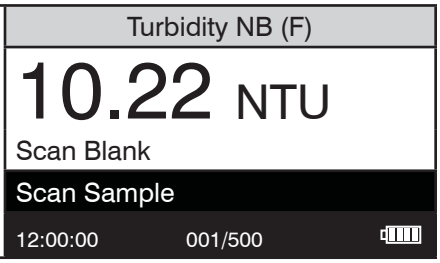
6. Wipe the tube thoroughly with a lint-free cloth.



7. Open the meter lid. Insert the tube into the chamber. Align the index line on the tube with the index arrow on the meter. Close the lid.



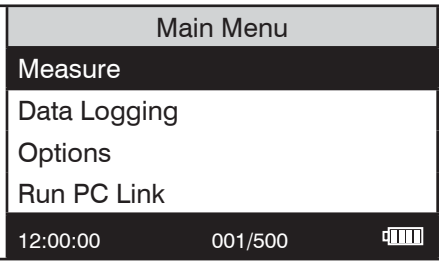
8. Press ENTER to select **Scan Sample**. The screen will display **Reading** for about 1 second. The result will appear on the screen.



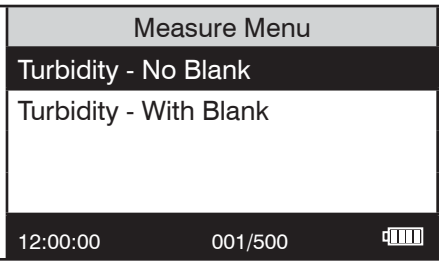
■ ANALYSIS WITH BLANKING PROCEDURE

To obtain the most accurate results the meter should be blanked before measuring a sample. The blanking step is not as critical for samples above 10 NTU. The meter should always be blanked before reading samples below 10 NTU.

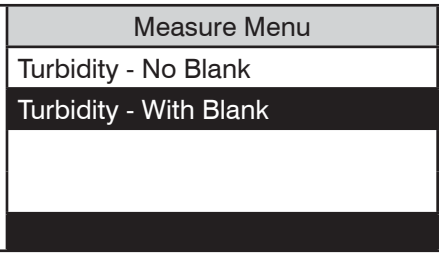
1. Press and briefly hold ⏻ to turn the meter on. The LaMotte logo screen will appear for about 3 seconds and the **Main Menu** will appear.



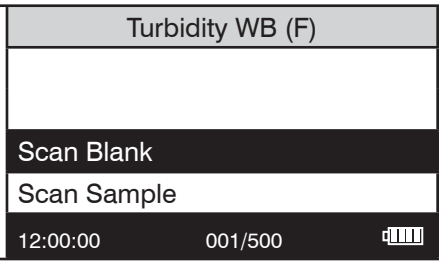
2. Press ENTER to select **Measure**.

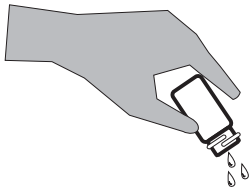
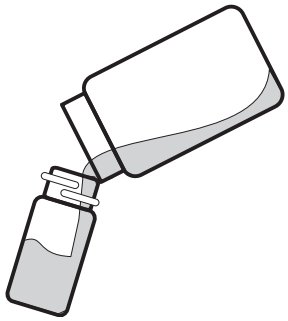
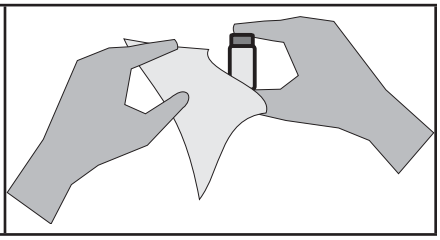
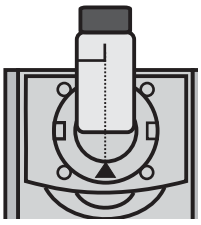


3. Press ⌄ to scroll to **Turbidity - With Blank**.

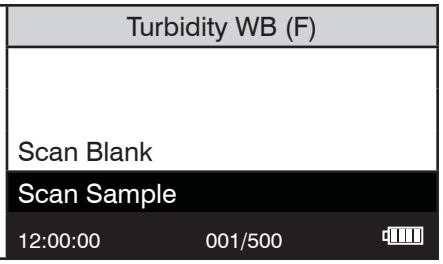


4. Press ENTER to select **Turbidity - With Blank**.

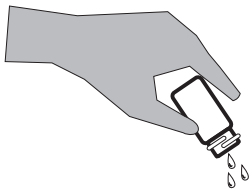


5. Rinse a clean tube (0290) three times with the blank. If samples are expected to read below 1 NTU the meter should be blanked with a 0 NTU Primary Standard or prepared turbidity-free (<0.1 NTU) water. For the most accurate results, use the same tube for the blank and the sample.	
6. Fill the tube to the fill line with the blank. Pour the blank down the inside of the tube to avoid creating bubbles. Cap the tube.	
7. Wipe the tube thoroughly with a lint-free cloth.	
8. Open the meter lid. Insert the tube into the chamber. Align the index line on the tube with the index arrow on the meter. Close the lid.	

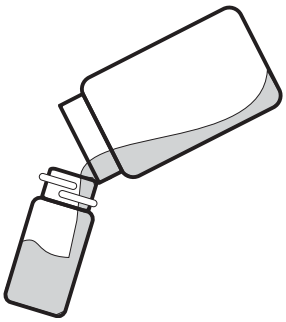
9. Press ENTER to scan the blank. The screen will display **Blank Done** for about 1 second and then return to the **Turbidity - With Blank** menu.



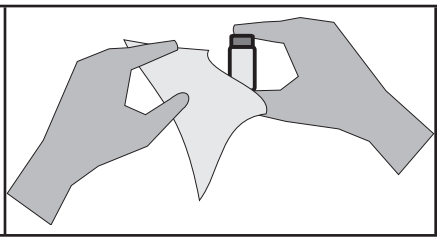
10. Rinse a clean tube (0290), or the same tube, three times with the sample.



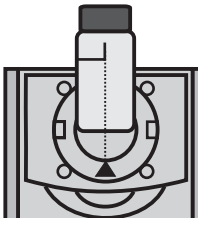
11. Fill the tube to the fill line with the standard. Pour the standard down the inside of the tube to avoid creating bubbles. Cap the tube.

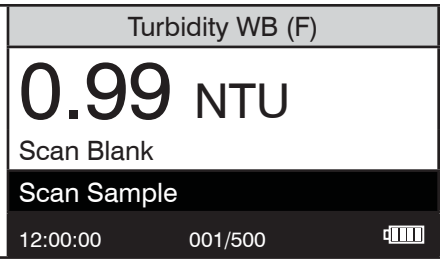


12. Wipe the tube thoroughly with a lint-free cloth.



13. Open the meter lid. Insert the tube into the chamber. Align the index line on the tube with the index arrow on the meter. Close the lid.



14. Press ENTER to scan the standard. The screen will display **Reading** for about 1 second. The result will appear on the screen.	

NOTE: The meter will remember the last scanned blank reading. It is not necessary to scan a blank each time the test is performed. To use the previous blank reading, instead of scanning a new one, scroll to Scan Sample and proceed. For the most accurate results, the meter should be blanked before each test and the same tube should be used for the blank and the reacted sample.

■ DILUTION PROCEDURES

If a sample is encountered that is more than 4000 NTU, a careful dilution with 0 NTU or very low turbidity water will bring the sample into an acceptable range. However, there is no guarantee that halving the concentration will exactly halve the NTU value. Particulates often react in an unpredictable manner when diluted.

Turbidity-Free Water

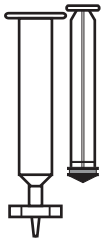
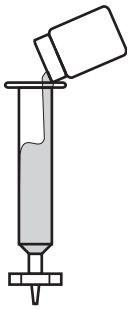
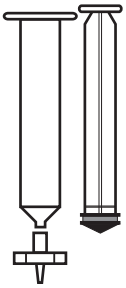
The definition of low turbidity and turbidity-free water has changed as filter technology has changed and nephelometric instruments have become more sensitive. At one time turbidity-free water was defined as water that had passed through a 0.6 micron filter. Now 0.1 micron filters are available and higher purity water is possible. Water that has been passed through a 0.1 micron filter could be considered particle free and therefore turbidity free, 0 NTU water. Turbidity is caused by scattered light. Therefore, low turbidity water is water without any particles that scatter a measurable amount of light. But water that passed through a 0.1 micron filter may still have detectable light scatter with modern instruments. This light scattering can be the result of dissolved molecules or sub-micron sized particles that can not be filtered out of the water. Because there may still be a small amount of scattered light from dissolved molecules, high purity water is often called low turbidity water and assigned a value of 0.01 or 0.02 NTU. However, because this water is used as a baseline to compare to sample water, the difference between the sample and the low turbidity or turbidity-free water will be the same whether it is called 0.00 NTU or 0.02 NTU. For design simplicity the 2020we/wi uses the term turbidity-free water and the value of 0.00 NTU.

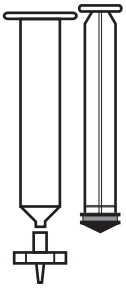
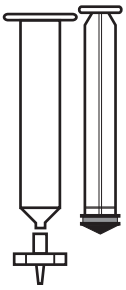
■ PREPARATION OF TURBIDITY-FREE WATER

A 0 NTU Standard (Code 1480) is included with the meter. An accessory package (Code 4185) is available for preparing turbidity-free water for blanking the meter and dilution of high turbidity samples.

The preparation of turbidity-free water requires careful technique.

Introduction of foreign matter will affect the turbidity reading. A filtering device with a special membrane filter is used to prepare turbidity-free water. The filter, filter holder and syringe must be conditioned by forcing at least two syringes full of deionized water through the filtering apparatus to remove foreign matter. The first and second rinses should be discarded. Turbidity-free water as prepared with the following procedure may be stored in the dark at room temperature in a clean glass bottle with a screw cap and used as required. The storage container should be rinsed thoroughly with filtered deionized water before filling. The water should be periodically inspected for foreign matter in bright light.

1. Remove the plunger from the syringe (0943). Attach the filter to the bottom of the syringe.	
2. Pour approximately 50 mL of deionized water into the barrel of the syringe. Insert the plunger. Exert pressure on the plunger to slowly force the water through the filter. Collect water in the clean storage container. Rinse walls of the container then discard this rinse water.	
3. Remove the filter from the syringe. Remove the plunger from the barrel. (This step is required to prevent rupturing the filter by the vacuum that would be created when the plunger is removed.)	

4. Replace the filter and repeat step 2 for a second rinse of the syringe and storage container.	
5. Remove the filter from the syringe. Remove the plunger from the barrel. Replace the filter and fill the syringe with approximately 50 mL of deionized water. Filter the water into the storage container and save this turbidity-free water.	
6. Repeat Step 5 until the desired amount of turbidity-free water has been collected.	

TESTING TIPS

1. Samples should be collected in a clean glass or polyethylene container.
2. Samples should be analyzed as soon as possible after collection.
3. Gently mix sample by inverting before taking a reading but avoid introducing air bubbles.
4. For the most precise results, follow the recommended procedure for wiping a filled tube before placing it in the meter chamber. Invert tube very slowly and gently three times to mix the sample. Surround the tube with a clean, lint-free cloth. Press the cloth around the tube. Rotate the tube in the cloth three times to assure that all areas of the tube have been wiped.
5. Discard tubes that have significant scratches and imperfections in the light pass zones. (Central zone between bottom and fill line).
6. When reading very low turbidity samples, do not use tubes or caps that have been used previously with high turbidity samples.
7. Use the averaging option for low level measurements of turbidity.

8. The meter should be placed on a surface that is free from vibrations. Vibrations can cause high readings.
9. Turbidity readings will be affected by electric fields around motors.
10. Carbon in the sample will absorb light and cause low readings.
11. Excessive color in a sample will absorb light and cause low readings. The user should verify if a certain level of color will cause a significant error at the level of turbidity being tested.
12. Observe shelf life recommendations for turbidity standards.
13. Do not use silicone oil on tubes when testing turbidity with the 2020we/wi.
14. When testing at low concentrations use the same tube for the blank and the sample.
15. Always insert tube into the meter chamber with the same amount of pressure and to the same depth.
16. Occasionally clean the chamber with a damp lint-free wipe, followed by a Windex® dampened wipe. A clean chamber and tubes are essential for reliable results.
17. For the greatest accuracy during the calibration procedure, be sure that after the meter is blanked and the blank is scanned as a sample, the reading is 0.00. If not, reblank the meter and scan the blank again until it reads 0.00. When scanning the calibration standards as the sample, scan the calibration standard three times removing the tube from the chamber after each scan. The readings should be consistent. Use the last consistent reading to calibrate the meter. If the readings are not consistent, avoid using an aberrant reading to calibrate the meter.
18. Calibrate the meter daily.
19. Calibrate the meter with a 1.0 NTU Standard if samples are expected to be 1.0 NTU or less. Calibrate the meter with a 10.0 NTU Standard if samples are expected to be 1.0 NTU or greater.

TROUBLESHOOTING GUIDE

■ TROUBLESHOOTING

PROBLEM	*REASON*	*SOLUTION*
“Blank?”	Sample is reading lower than the blank.	With samples of very low concentration reblank or record as zero. On samples of higher concentration reblank and read again.
Flashing	Low battery. Readings are reliable.	Charge battery or use USB wall/computer charger.
“Low Battery”	Battery voltage is very low. Readings are not reliable.	Charge battery or use USB wall/computer charger.
“Shut Down Low Batt” Shut Down	Battery is too low to operate the unit.	Charge battery or use USB wall/computer charger.
“Over range”	Sample is outside of acceptable range.	Dilute sample and test again.
“Error1”	High readings with 90° and 180° detectors.	Dilute sample by at least 50% and retest.
Lost in meter menus	Reset to factory default settings.	Follow Procedure on page 9 or page 26.
Unusually large negative or positive readings when performing calibration	Incorrect standards used to calibrate meter.	Use fresh 0.0 standard in clean tube. Reset meter to factory default settings. Recalibrate meter.

■ STRAY LIGHT

The accuracy of readings on the 2020we/wi should not be affected by stray light. Make sure that the sample compartment lid is always fully closed when taking readings. The backlight will interfere with turbidity readings. The meter will temporarily disable the backlight while turbidity measurements are being taken.

GENERAL OPERATING INFORMATION

■ OVERVIEW

The 2020we/wi is a portable, microprocessor controlled, direct reading nephelometer. Turbidity is measured directly by either EPA Method 180.1 or ISO Method 7027. It has a graphical liquid crystal display and 6 button keypad. These allow the user to select options from the menu driven software, to directly read test results or to review stored results of previous tests in the data logger. The menus can be displayed in seven different languages.

The 2020we/wi uses a state of the art, multi-detector optical configuration that assures long term stability of calibrations, high precision and accuracy and low detection limits. All readings are determined by sophisticated digital signal processing algorithms, minimizing fluctuations in readings and enabling rapid, repeatable measurements. The microprocessor and optics enable a dynamic range and auto-ranging over several ranges. Energy efficient LED light sources are used for ISO turbidity. EPA turbidity uses a tungsten filament light source that meets or exceeds EPA specifications and is designed for a uniform light spot image and stable output.

A USB computer/wall charger or Lithium battery powers the 2020we/wi.

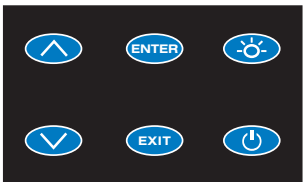
A USB port on the back of the meter allows an interface of the meter with a Windows-based computer for real-time data acquisition and data storage using a PC. The 2020we/wi may be interfaced with any Windows-based computer by using the LaMotte SMARTLink 3 Program.

GENERAL OPERATING INFORMATION

The operation of the 2020we/wi is controlled by the menu driven software and user interface. A menu is a list of choices. This allows a selection of various tasks for the 2020we/wi to perform, such as, scan blank and scan sample. The keypad is used to make menu selections that are viewed on the display.

■ The Keypad

	This button will scroll up through a list of menu selections.
	The button is used to select choices in a menu viewed in the display.
	This button controls the backlight on the display.
	This button will scroll down through a list of menu selections.
	This button exits to the previous menu.
	This button turns the meter on or off.

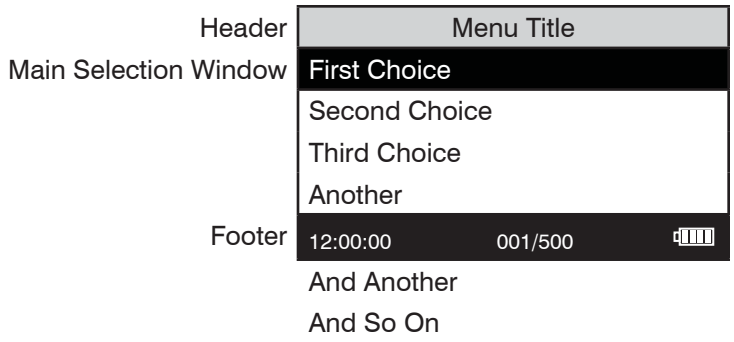


■ THE DISPLAY & MENUS

The display allows menu selections to be viewed and selected. These selections instruct the 2020we/wi to perform specific tasks. The menus are viewed in the display using two general formats that are followed from one menu to the next. Each menu is a list of choices or selections.

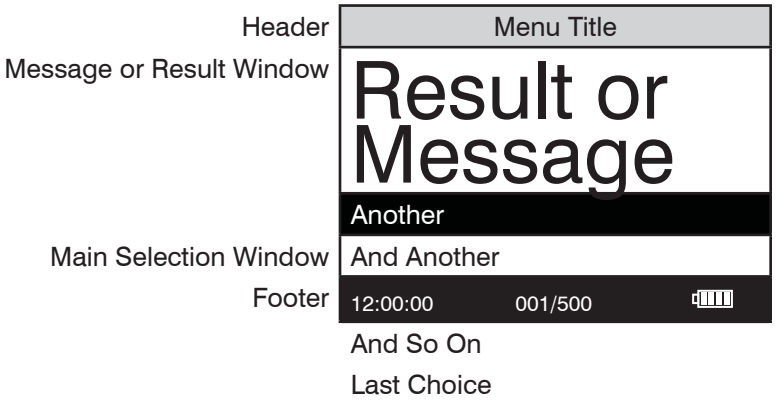
The display has a header line at the top and a footer line at the bottom. The header displays the title of the current menu. The footer line displays the time and the date, the data logger status and the battery status. The menu selection window is in the middle of the display between the header and the footer.

The menu selection window displays information in two general formats. In the first format only menu selections are displayed. Up to 4 lines of menu selections may be displayed. If more selections are available they can be viewed by pressing the arrow buttons to scroll the other menu selections into the menu selection window. Think of the menu selections as a vertical list in the display that moves up or down each time an arrow button  is pressed. Some menus in the 2020we/wi are looping menus. The top and bottom menu choices are connected in a loop. Scrolling down past the bottom of the menu will lead to the top of the menu. Scrolling up past the top of the menu will lead to the bottom of the menu.



A black bar will indicate the menu choice. As the menu is scrolled through, the black bar will highlight different menu choices. Pressing the ENTER button will select the menu choice that is indicated by the black bar.

In the second format the menu choice window takes advantage of the graphical capabilities of the display. Large format graphic information, such as test results or error messages or the LaMotte logo is displayed. The top two lines of the display are used to display information in a large, easy to read format. The menus work in the same way as previously described but two lines of the menu are visible at the bottom of the display.



As described previously, the EXIT button allows an exit or escape from the current menu and a return to the previous menu. This allows a rapid exit from an inner menu to the main menu by repeatedly pushing the EXIT button. Pushing ⏻ at any time will turn the 2020we/wi off.

The display may show the following messages:

	Battery Status
	More choices are available and can be viewed by scrolling up and/or down through the display.
Header	Identifies the current menu and information on units and reagent systems if applicable.
Footer	In the data logging mode the number of the data point is displayed and the total number of data points in the memory will be shown. The footer also shows current time and battery status

■ NEGATIVE RESULTS

There are always small variations in readings with analytical instruments. Often these variations can be observed by taking multiple readings of the same sample. These variations will fall above and below an average reading. Repeated readings on a 0.00 sample might give readings above and below 0.00. Therefore, negative readings are possible and expected on samples with concentrations at or near zero. This does not mean there is a negative concentration in the sample. It means the sample reading was less than the blank reading. Small negative readings can indicate that the sample was at or near the detection limit. This is a normal variation that results in a negative reading. A large negative reading, however, is not normal and indicates a problem. Some instruments are designed to display negative readings as zero. In this type of instrument, if the meter displayed zero when the result was actually a large negative number there would be no indication that a problem existed. For this reason, the 2020we/wi displays negative numbers for turbidity.

■ TUBES

The 2020we/wi uses one type of tube (Code 0290). There is no need for a special turbidity tube.

The handling of the tubes is of utmost importance. Tubes must be clean and free from lint, fingerprints, dried spills and significant scratches, especially the central zone between the bottom and the sample line.

Scratches, fingerprints and water droplets on the tube can cause stray light interference leading to inaccurate results when measuring turbidity. Scratches and abrasions will affect the accuracy of the readings. Tubes that have been scratched in the light zone through excessive use should be discarded and replaced with new ones.

Tubes should always be washed on the inside and outside with mild detergent prior to use to remove dirt or fingerprints. The tubes should be

allowed to air-dry in an inverted position to prevent dust from entering the tubes. Dry tubes should be stored with the caps on to prevent contamination.

After a tube has been filled and capped, it should be held by the cap and the outside surface should be wiped with a clean, lint-free absorbent cloth until it is dry and smudge-free. Handling the tube only by the cap will avoid problems from fingerprints. Always set the clean tube aside on a clean surface that will not contaminate the tube. It is imperative that the tubes and light chamber be clean and dry. The outside of the tubes should be dried with a clean, lint-free cloth or disposable wipe before they are placed in the meter chamber.

Tubes should be emptied and cleaned as soon as possible after reading a sample to prevent deposition of particulates on the inside of the tubes. When highly accurate results are required, reduce error by designating tubes to be used only for very low turbidity and very high turbidity testing.

Variability in the geometry of the glassware and technique is the predominate cause of variability in results. Slight variations in wall thickness and the diameter of the tubes may lead to slight variations in the test results. To eliminate this error the tubes should be placed in the chamber with the same orientation each time.

COMPUTER CONNECTION

■ PC LINK

The 2020we/wi may be interfaced with any Windows-based computer by using the LaMotte SMARTLink 3 Program and USB Cable. The program will store test information and results in a database.

■ OUTPUT

USB

■ COMPUTER CONNECTION

USB Type A, USB mini B, Order Cable Code 1720.

BATTERY OPERATION

The 2020we/wi may be operated on battery power or using a computer/AC wall adapter. If using the meter as a bench top unit, use the AC wall adapter if possible to extend the battery life. The meter will remain on when the USB adapter is used.

The battery icon will show no bars and flash when the unit first turns on. Then the indicator will indicate the battery status by showing 0, 1, 2, 3 or 4 bars.

It will take 5 hours to fully change a low battery. The battery icon will flash when the battery is charging. The battery icon will show four bars and stop flashing when it is fully charged. The charging circuit will automatically switch to a float charge when the battery is fully charged. The charger may remain connected. Some computers will NOT supply

power to their USB ports during standby operation. The wall charger will charge the unit continuously.

The battery icon will show no bars and continuously flash if the battery is getting low but the unit will still operate normally. A “Low Battery” message on the status bar of the display will replace the time when the battery voltage is too low for proper operation and accuracy may be degraded. A “Shutdown Low Batt” message on the display will appear for a few seconds before the power is switched off when the battery is too low to operate the unit.

To extend the battery life:

- Shut down the unit with the power switch when not taking measurements or use the power save option to have the unit automatically turn off after 5 minutes.
- Store the unit in a cool dry place.
- Fully charge the battery before storing the unit for extended periods of time.
- Limit backlight use. The unit consumes 3X normal power with the backlight on. Set the backlight time option to 10 seconds, or select “Button Control” and keep the backlight off.

MAINTENANCE

■ CLEANING

Clean the exterior housing with a damp, lint-free cloth. Do not allow water to enter the light chamber or any other parts of the meter. To clean the light chamber and optics area, point a can of compressed air into the light chamber and blow the pressurized air into the light chamber. Use a cotton swab dampened with Windex® window cleaner to gently swab the interior of the chamber. Do not use alcohol; it will leave a thin residue over the optics when dry.

■ REPAIRS

Should it be necessary to return the meter for repair or servicing, pack the meter carefully in a suitable container with adequate packing material. A return authorization number must be obtained from LaMotte Company by calling 800-344-3100 (US only) or 410-778-3100, faxing 410-778-6394, or emailing tech@lamotte.com. Often a problem can be resolved over the phone or by email. If a return of the meter is necessary, attach a letter with the return authorization number, meter serial number, a brief description of problem and contact information including phone and FAX numbers to the shipping carton. This information will enable the service department to make the required repairs more efficiently.

■ METER DISPOSAL

Waste Electrical and Electronic Equipment (WEEE)

Natural resources were used in the production of this equipment. This equipment may contain materials that are hazardous to health and the environment. To avoid harm to the environment and natural resources, the use of appropriate take-back systems is recommended. The crossed out wheeled bin symbol on the meter encourages the use of these systems when disposing of this equipment.

Take-back systems will allow the materials to be reused or recycled in a way that will not harm the environment. For more information on approved collection, reuse, and recycling systems contact local or regional waste administration or recycling services.

GENERAL INFORMATION

■ PACKAGING AND DELIVERY

Experienced packaging personnel at LaMotte Company assure adequate protection against normal hazards encountered in transportation of shipments.

After the product leaves LaMotte Company, all responsibility for safe delivery is assured by the transportation company. Damage claims must be filed immediately with the transportation company to receive compensation for damaged goods.

■ GENERAL PRECAUTIONS

READ THE INSTRUCTION MANUAL BEFORE ATTEMPTING TO SET UP OR OPERATE THE METER. Failure to do so could result in personal injury or damage to the meter. The meter should not be used or stored in a wet or corrosive environment. Care should be taken to prevent water from wet tubes from entering the meter chamber.

NEVER PUT WET TUBES IN THE METER.

■ SAFETY PRECAUTIONS

Read the label on all reagent containers. Some labels include precautionary notices and first aid information. Certain reagents are considered potential health hazards and are designated with a * in the instruction manual. To view or print a Material Safety Data Sheet (MSDS) for these reagents go to www.lamotte.com. To obtain a printed copy, contact LaMotte by e-mail, phone or FAX. Additional information for all LaMotte reagents is available in the United States, Canada, Puerto Rico, and the US Virgin Islands from Chem-Tel by calling 1-800-255-3924. For other areas, call 813-248-0585 collect to contact Chem-Tel's International access number. Each reagent can be identified by the four-digit number listed on the upper left corner of the reagent label, in the contents list and in the test procedures.

■ LIMITS OF LIABILITY

Under no circumstances shall LaMotte Company be liable for loss of life, property, profits, or other damages incurred through the use or misuse of their products.

■ SPECIFICATIONS - 2020we/wi

Instrument Type:	Nephelometer
Standard:	EPA 180.1, 2020we; ISO7027, 2020wi
Units of Measure:	NTU (Nephelometric Turbidity Units) (2020we only) FNU (Formazin Nephelometric Units) (2020wi only) ASBC (American Society of Brewing Chemists) EBC (European Brewery Convention)
Range:	0-4000 NTU, 0-4000 FNU, 0-10,500 ASBC, 0-150 EBC
Range Selection:	Automatic
Resolution: (display)	0.01 NTU, 0–10.99 NTU Range 0.1 NTU, 11.0–109.9 NTU Range 1 NTU, 110–4000 NTU Range
Accuracy:	From 0-2.5 NTU the accuracy is ±0.05 NTU. From 2.5-100 NTU the accuracy is ±2%. Above 100 NTU the accuracy is ±3%.
Detection Limit:	0.05 NTU
Light Source:	Tungsten lamp 2300°C ±50 °C, 2020we; IR LED 850 nm ±10 nm, spectral bandwidth 50 nm, 2020wi
Detector	Photodiode, centered at 90°, maximum peak 400-600 nm, TC-3000we Photodiode, centered at 90°, TC-3000wi
Response Time:	<2 seconds
Signal Averaging:	Yes
Sample Chamber:	Accepts 25 mm flat-bottomed test tubes
Sample:	10 mL in capped tube
Display:	Graphic Liquid Crystal Display
Software:	*Auto Shut-off:* 5, 10, 30 min, disabled *Calibration:* Field adjustable, 2-points *Data Logging:* 500 points
Languages:	English, Spanish, French, Portuguese, Italian, Chinese, Janpanese (Kana)
Temperature:	Operation: 0–50 °C; Storage: -40–60 °C

Operation Humidity Range:	0–90 % RH, non-condensing
Auto Shut-off:	5, 10, 30 min, disabled
Waterproof:	IP67
Power Source†:	USB computer/wall charger or Lithiun ion rechargeable battery 2200 mAH, 3.7V
Battery Life:	~380 tests (backlight on) to 1000 tests (backlight off) (with signal averaging disabled)
Dimensions:	(W x L x H) 8.84 x 19.05 x 6.35 cm; 3.5 x 7.5 x 2.2 inches
Weight:	362 g, 13 oz (meter only)
USB Interface	mini B

†CE Mark: The device complies to the product specifications for the Low Voltage Directive.

■ STATISTICAL & TECHNICAL DEFINITIONS RELATED TO PRODUCT SPECIFICATIONS

Method Detection Limit (MDL): "The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte."[1] Note that, "As Dr. William Horwitz once stated, 'In almost all cases when dealing with a limit of detection or limit of determination, the primary purpose of determining that limit is to stay away from it.'"[2]

Accuracy: Accuracy is the nearness of a measurement to the accepted or true value.[3] The accuracy can be expressed as a range, about the true value, in which a measurement occurs (i.e. ±0.5 ppm). It can also be expressed as the % recovery of a known amount of analyte in a determination of the analyte (i.e. 103.5 %).

Resolution: Resolution is the smallest discernible difference between any two measurements that can be made.[4] For meters this is usually how many decimal places are displayed. (i.e. 0.01). Note that the resolution many change with concentration or range. In some cases the resolution may be less than the smallest interval, if it is possible to make a reading that falls between calibration marks. A word of caution, that resolution has very little relationship to accuracy or precision. The resolution will always be less than the accuracy or precision but it is not a statistical measure of how well a method of analysis works. The resolution can be very, very good and the accuracy and precision can be very bad! This is not a useful measure of the performance of a test method.

Repeatability: Repeatability is the within-run precision.[5] A run is a

single data set, from set up to clean up. Generally, one run occurs on one day. However, for meter calibrations, a single calibration is considered a single run or data set, even though it may take 2 or 3 days.

Reproducibility: Reproducibility is the between-run precision.[6]

Detection Limit (DL): The detection limit (DL) for the 2020we/wi is defined as the minimum value or concentration that can be determined by the meter, which is greater than zero, independent of matrix, glassware, and other sample handling sources of error. It is the detection limit for the optical system of the meter.

[1] CFR 40, part 136, appendix B

[2] Statistics in Analytical Chemistry: Part 7 – A Review, D. Coleman and L Vanatta, American Laboratory, Sept 2003, P. 31.

[3] Skoog, D.A., West, D. M., *Fundamental of Analytical Chemistry*, 2nd ed., Holt Rinehart and Winston, Inc, 1969, p. 26.

[4] Statistics in Analytical Chemistry: Part 7 – A Review, D. Coleman and L Vanatta, American Laboratory, Sept 2003, P. 34.

[5] Jeffery G. H., Basset J., Mendham J., Denney R. C., *Vogel's Textbook of Quantitative Chemical Analysis*, 5th ed., Longman Scientific & Technical, 1989, p. 130.

[6] Jeffery G. H., Basset J., Mendham J., Denney R. C., *Vogel's Textbook of Quantitative Chemical Analysis*, 5th ed., Longman Scientific & Technical, 1989, p. 130

■ CONTENTS & ACCESSORIES

	2020we Kit EPA Version Code 1970-EPA	2020wi Kit ISO Version Code 1970-ISO
Contents	*Code*	*Code*
0 NTU Standard, 60 mL	1480	1480
1 NTU Standard, 60 mL	1450	1453
10 NTU Standard, 60 mL	1451	1454
Water Sample Bottle, 60 mL	0688	0688
Tubes, 4	—	—
Cable, USB, 3 ft.	1720	1720
USB Wall Plug	1721	1721

Accessories	
Code	*Description*
1452	100 NTU Standard, 60 mL (EPA)
1455	100 NTU Standard, 60 mL (ISO)
0290-6	Tubes, Code 0290, Set of 6
4185	Turbidity-Free Water Kit
2-2097	Filters, 0.1 micron, Pack of 50
1901-CD	SMARTLink 3 Software

■ EPA COMPLIANCE

The 2020we meter meets or exceeds EPA design specifications for NPDWR and NPDES turbidity monitoring programs as specified by the USEPA method 180.1.

■ ISO Compliance

This 2020wi meter meets or exceeds ISO design criteria for quantitative methods of turbidity using optical turbidimeters as specified by ISO 7027.

■ CE COMPLIANCE

The 2020we and 2020wi meters have been independently tested and have earned the European CE Mark of compliance for electromagnetic compatibility and safety. To view certificates of compliance, go to the LaMotte website at www.lamotte.com.

NOTE: The device complies to the product specifications for the Low Voltage Directive.

■ WARRANTY

LaMotte Company warrants this instrument to be free of defects in parts and workmanship for 2 years from the date of shipment. If it should become necessary to return the instrument for service during or beyond the warranty period, contact our Technical Service Department at 1-800-344-3100 for a return authorization number or visit www.lamotte.com for troubleshooting help. The sender is responsible for shipping charges, freight, insurance and proper packaging to prevent damage in transit. This warranty does not apply to defects resulting from action of the user such as misuse, improper wiring, operation outside of specification, improper maintenance or repair, or unauthorized modification. LaMotte Company specifically disclaims any implied warranties or merchantability or fitness for a specific purpose and will not be liable for any direct, indirect, incidental or consequential damages. LaMotte Company's total liability is limited to repair or replacement of the product. The warranty set forth above is inclusive and no other warranty, whether written or oral, is expressed or implied.

802 Washington Ave • Chestertown • Maryland • 21620 • USA
410-778-3100 • 800-344-3100
www.lamotte.com

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Appendix G

Field Variance Form

FIELD VARIANCE FORM

DATE:	02 May 2017	LOCATION:	Seneca Army Depot Activity
TASK ORDER:	W912DY-09-D-0062-0023	PROJECT NAME:	PFAS Site Investigation

The following is for the review and subsequent approval of a variance to the subject project approved work plan and QAPP. This variance, as described below, is a result of site conditions encountered during the field program.

PROCEDURE AS IT APPEARS IN THE APPROVED WP/QAPP:

The general scope of the activities related to sampling for PFAS at SEAD-26 and SEAD-122E are to:

- Collect groundwater grab samples within the upper till/weathered shale aquifer using direct push technology (DPT) drilling techniques; and
- Analyze samples for PFAS.

Drilling shall be performed using a truck-mounted drill rig using DPT techniques and in accordance with *SOP #4 – Monitoring Well Installation*. Due to the tight formation, the groundwater at Seneca moves slowly and wells often recharge very slowly. Due to slow groundwater infiltration and the site stratigraphy, and input from NYSDEC and EPA, temporary monitoring wells will be installed for groundwater sampling.

Summary of SOP#3 – Groundwater Sample Collection

The following procedure will be followed for the collection of groundwater samples:

- Record comments pertinent to the color and any obvious odors associated with the water.
- Arrange and label necessary sample bottles and ensure that preservatives are added, as required. Labeling must include a unique sample number, date of sampling, the initials of the sampling personnel, and the identity of the sample fraction. Additionally, provide any information pertinent to the preservation materials or chemicals used in the samples.
- Samples shall be collected using high density polyethylene (HDPE) tubing. Purging can be performed either by fitting the tubing with a check valve and manually pumping or by using a peristaltic pump.
- Immediately seal each sample bottle, double bag it using Zip-Lock® brand bags and place the sample bottles on ice in a cooler to maintain sample integrity. Do not allow the samples to freeze, as the bottles may break.
- Once sampling is completed, recover sample tubing and dispose;
- Clean up and remove any debris left from the sampling event. Be sure that wastes are properly containerized and labeled.
- Review sampling records. Ensure that necessary data is completed. Add additional information as may be needed.

RECOMMENDED CHANGE TO THE ABOVE PROCEDURE:

Drilling activities at SEAD 122 and SEAD 26 indicate that subsurface conditions at both sites are composed of dry, tight glacial till on top of shale bedrock. In general, bedrock was encountered at a depth of 12 feet below ground surface (bgs). Within a majority of the bore holes, the drilling team only encountered moist conditions in small seams of sand and gravel within the till. Due to the lack of recharge within the wells, the decision was

made to postpone the PFAS groundwater sampling at SEAD 122 and SEAD 26 for a minimum of two weeks instead of collecting the samples immediately after well installation to allow for water to infiltrate the monitoring wells.

Due to the lack of recharge within the wells, when drilling was completed, the temporary wells were carefully gauged (i.e., only exposing the groundwater to the stainless-steel portion of a properly decontaminated water gauge). The field team leader reported limited saturated thickness within many of the wells. The wells at SEAD 26 contained the least amount of groundwater within saturated thicknesses of less than 3 feet.

During the postponement of the groundwater sampling, the groundwater sampling methodology was reviewed given the lack of water column available and the slow recharge of the formation. Two issues were discussed: 1) Would the limited saturated thickness provide sufficient groundwater to collect a sample and submit to the lab for analysis, and 2) Would the peristaltic pump be capable of extracting water out of a well with minimal saturated thickness or would it evacuate the well so quickly that we would have to wait for the well to recharge.

On 28 March 2017, the Army, EPA, and NYSDEC were notified via email of these potential concerns. In the email, the primary field strategy was confirmed as use of the peristaltic pump to collect the groundwater sample; however, if there was a minimal amount of saturated thickness in the well the use of a stainless-steel bailer was proposed by the Army. NYSDEC and USEPA expressed concerns about the use of bailer and cross contamination that could be introduced by the check ball.

A teleconference was held on 01 May 2017 between NYSDEC and Parsons to discuss the sampling approach. Parsons agreed to not use a bailer at any of the PFAS sites due to potential cross-contamination from the plastic check valve balls in the stainless-steel bailers. Every effort will be made to collect the groundwater samples using the peristaltic pump. It was agreed that the wells would not be purged and the groundwater would be collected as grab samples. Additionally, Parsons was informed by the lab that a minimum of 250mL (one sample bottle) could be submitted without an increase in the reporting limits. If 250mL could not be extracted from the well, a smaller sample would still be sent to the lab; however, the results would include a corresponding increase in the reporting limit.

		SIGNATURE	
		APPROVED	REJECTED
Parsons Project Manager:	Beth Badik		
USACE CENAN Project Manager	Randy Battaglia	BATTAGLIA.RANDALL.W.1228816724 Digitally signed by BATTAGLIA.RANDALL.W.1228816724 DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USA, cn=BATTAGLIA.RANDALL.W.1228816724 Date: 2017.05.03 13:10:57 -04'00'	
USACE Project Manager	Derek Pommerenck	POMMERENCK.DEREK.ANDREW.1080769748 Digitally signed by POMMERENCK.DEREK.ANDREW.1080769748 DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USA, cn=POMMERENCK.DEREK.ANDREW.1080769748 Date: 2017.05.10 11:15:12 -05'00'	