DRAFT
SAMPLING AND ANALYSIS PLAN
SENECA ARMY DEPOT ACTIVITY
ROMULUS, NEW YORK

May 20, 2005

Prepared by:

PARSONS
150 Federal Street
Boston, MA 02110
617-946-9400

EPA SITE ID# NY0213820830
NY SITE ID# 8-50-006
AFCEE Contract Number FA8903-04-D-8675
DELIVERY ORDER NO. 0012
CRDL A004

Reviewed and Approved by:

_____ _____
Todd Heino, Parsons Project Manager, Date

_____ _____
James Lowerre, Parsons Quality Assurance Officer Date

_____ _____
Katherine Lapierre, Parsons Project Chemist Date

_____ _____
Julio F. Vazquez, USEPA Region 2 Project Manager Date

_____ _____
Kuldeep K. Gupta, NYSDEC Project Manager Date

_____ _____
Lonnie Wolfe, AFCEE Contracting Officer's Representative Date

TABLE OF CONTENTS

LIST OF APPENDICES

ACRONYMS

A2LA	American Association of Laboratory Accreditation
AFCEE	Air Force Center for Environmental Excellence
AL	Action Limit
ARAR	Applicable or Relevant and Appropriate Requirement
ASP	Analytical Service Protocol
ASTM	American Society for Testing and Materials
bgs	Below Ground Surface
BRAC	Base Realignment and Closure
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulation
CCV	Continuing Calibration Verification
CLP	Contract Laboratory Program
COC	Chain-Of-Custody
COR	Contracting Officer's Representative
CRQL	Contract Required Quantitation Limit
CSM	Conceptual Site Model
DEQPPM	Defense Environmental Quality Program Policy Memorandum
DER	Division of Environmental Remediation
DNAPL	Dense Non-Aqueous Phase Liquid
DO	Dissolved Oxygen
DOA	Department of the Army
DoD	Department of Defense
DOT	Department of Transportation
DQCR	Daily Quality Control Report
DQI	Data Quality Indicator
DQO	Data Quality Objectives
DQOP	Data Quality Objectives Process
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Approval Program
EO	Executive Order
EPA	(US) Environmental Protection Agency
ERPIMS	Environmental Restoration Program Information Management System
FB	Field Blank
FFA	Federal Facilities Agreement
FID	Flame Ionization Detector
FS	Feasibility Study
FSP	Field Sampling Plan
ft	Feet or foot
GALP	Good Automated Laboratory Practices
GC	Gas Chromatography
GPR	Ground Penetrating Radar
HRR	Historical Records Review
HSP	Health and Safety Plan
HTRW	Hazardous, Toxic and Radioactive Waste
HPLC	High Performance Liquid Chromatography

IAW	in accordance with
lbs/gal	pounds per gallon
IC	Ion Chromatography
ICAL	Initial Calibration
ICS	Interference Check Sample
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectra
ICP-MS	Inductively Coupled Plasma-Mass Spectra
ID	Identification
IDL	Instruction Detection Limit
IDW	Investigation Derived Waste
LNAPL	Light Non-Aqueous Phase Liquid
IRP	Installation Restoration Program
IS	Internal Standard
LCS	Laboratory Control Sample
MB	Matrix Blank
MC	Munitions Constituents
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MEC	Munitions and Explosives of Concern
mL	milliliter
mL/L	milliliters per liter
MMRP	Military Munitions Restoration Program
MS	Mass Spectrometry/Matrix Spike
MSD	Matrix Spike Duplicate
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NCP	National Contingency Plan
NELAP	National Environmental Accreditation Program
NFA	No Further Action
NGVD	National Geodetic Vertical Datum
NIST	National Institute Standards and Technology
NPL	National Priorities List
NTU	Nephelometric Turbidity unit
NY	New York
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
OD	Outside Diameter
OSHA	Occupational Safety and Health Administration
OVA	Organic Vapor Analyzer
PAB	Project Analytical Batch
PAH	Polynuclear Aromatic Hydrocarbon
PARCC	Precision, Accuracy, Representativeness, Completeness, Comparability
PCB	Polychlorinated Biphenyl
PE	Performance Evaluation
PID	Photoionization Detector
PM	Project Manager
ppb	parts-per-billion
PPE	Personal Protective Equipment
ppm	parts-per-million
PQL	Practical Quantitation Limit

PQO	Project Quality Objectives
PVC	Polyvinyl Chloride
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Program Plan
QA/QC	Quality Assurance/ Quality Control
QC	Quality Control
QCSR	Quality Control Summary Report
QL	Quantitation Limit
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RIC	Reconstructed Ion Chromatograms
RI/FS	Remedial Investigation/Feasibility Study
RL	Reporting Limit
RQD	Rock Quality Designation
RPD	Relative Percent Difference
RT	Retention Time
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SB	Soil Boring
SCG	Standards, Criteria, and Guidance
SD	Sediment
SDG	Sample Delivery Group
SEDA	Seneca Army Depot Activity
SI	Site Inspection
SOP	Standard Operating Procedures
SOW	Statement of Work
SP	Spontaneous Potential
SPP	Systematic Planning Process
SQL	Sample Quantitation Limit
SS	Surface Soil
SSC	Site Safety Coordinator
SS-WP	Site Specific Work Plan
STARS	Spill Technology and Remediation Series
SVOC	Semivolatile Organic Compound
SW	Surface Water
SWMU	Solid Waste Management Unit
TAGM	Technical and Administrative Guidance Memorandum
TB	Trip Blank
TBC	To Be Considered
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively identified compound
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TOGS	Technical Operating Guidance Series
TP	Test Pit
TPH	Total Petroleum Hydrocarbon
TPP	Technical Project Planning

ug/Kg	micrograms per kilogram
ug/L	micrograms per liter
U.S.	United States
USACE	United States Army Corps of Engineers
USAESCH	US Army Engineering and Support Center, Huntsville
USCS	Unified Soil Classification System
USEPA	United States Environmental Protection Agency
USGS	U.S. Geological Survey
UXO	Unexploded Ordnance
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound
VTSR	verified time of sample receipt
WB	Wash or Rinse Blank
WP	Work Plan

1.0 INTRODUCTION

Parsons has prepared the Sampling and Analysis Plan (SAP) for the Seneca Army Depot Activity (SEDA) in Romulus, New York. This generic SAP will serve as an umbrella document under which project-specific tasks are conducted. Project-specific information is not covered in this generic SAP but is documented in detailed project-specific work plans, which use the generic SAP as an informational reference whenever appropriate. The use of this generic SAP, with supplemental project-specific work plans as needed, is a significant opportunity to use a graded approach, reducing repetition and streamlining the SAP development, review, and approval process.

The SAP consists of two parts: Quality Assurance Program Plan (QAPP, Sections 2 through 15) and Field Sampling Plan (FSP, Section 16).

The generic QAPP prepared for the Seneca Army Depot Activity states the expectations and specifications for obtaining the type and quality of environmental data needed for the project and describes the policies and procedures for ensuring that work process, products, or services satisfy the stated expectations and specifications. The QAPP includes definitions and generic goals for data quality and minimum requirements for quality assurance/ quality control (QA/QC) samples. The FSP provides general information and standard operating procedures (SOPs) applicable to sampling and analytical activities to be performed at all sites at SEDA.

It should be noted that the SAP may include discussions on procedures or methods that are not applicable to a specific site since it is intended to encompass all sites at the Seneca Depot. A Site-Specific work plan (SS-WP) will be prepared for each individual site where sampling and analytical activities are being conducted. The work plan will serve as addendums to this SAP. It is intended that once the SAP is finalized, it will not be modified (except for programmatic changes) and will serve as a programmatic document. Site-specific sampling information and any exceptions or proposed changes to the SAP will be addressed and included in the site-specific work plan. The majority of information contained in this SAP should not be repeated in the SS-WP. The methods specific to each site should specify the appropriate detection limit and reporting limit information. Any deviations from this SAP (e.g., holding times, detection limits, sampling methods, etc.) should be brought to the attention of the management team.

The SS-WP should not be a stand-alone document from this SAP. The SAP will provide the majority of the QA/QC information; the SS-WP should simply supplement this information by providing site-specific requirements.

The Seneca Site-Wide SAP is prepared consistent with the guidance including, but not limited to, the following:

- Guidance for Contract Deliverables, Appendix C: Quality Assurance Project Plan (QAPP), Version 4.0**,** Air Force Center for Environmental Excellence (AFCEE), 2005

- Guidance for Contract Deliverables, Appendix B: Model Field Sampling Plan, Version 1.2, AFCEE, 2002

- Guidance for the Data Quality Objectives Process, United States Environmental Protection Agency (USEPA) QA/G-4, 2000
- EPA Requirements for Quality Assurance Project Plans, USEPA QA/R-5, 2001
- Data Quality Objectives Process for Hazardous Waste Site Investigations, USEPA QA/G-4HW, 2000
- Uniform Federal Policy for Quality Assurance Project Plans, Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs, USEPA, 2004
- Guidance for Quality Assurance Project Plans, USEPA QA/G-5, 2002
- Quality Management Plan for Western Ecology Division, USEPA, 2001
- Guidance for the Development of Quality Assurance Project Plans for Environmental Monitoring Projects, USEPA Region 2, 2004
- Analytical Service Protocols, New York State Department of Environmental Conservation (NYSDEC), 2000
- Technical Guidance for Site Investigation and Remediation SW-96-09: Development and Review of Site Analytical Plans, NYSDEC, 2001
- Draft DER-10 Technical Guidance for Site Investigation and Remediation, NYSDEC, 2002
- Chemical Quality Assurance for Hazardous, Toxic and Radioactive Waste (HTRW) Project, United States Army Corps of Engineers (USACE) EM200-1-6, 1997
- Requirements for the Preparation of Sampling and Analysis Plan, USACE EM200-1-3, 2001

Appendix A presents a cross reference table for selected applicable SAP guidance.

2.0 PROJECT DESCRIPTION

2.1 SENECA ARMY DEPOT PROJECT BACKGROUND

SEDA is located approximately 40 miles south of Lake Ontario, near Romulus, New York (**Figure 1**). The Depot lies immediately west of the village of Romulus, New York (NY), 12 miles south of the villages of Waterloo and Seneca Falls, and 2.5 miles north of the village of Ovid, NY. The two closest major cities are Rochester, NY, which is located approximately 60 miles northwest, and Syracuse, NY, which is located approximately 60 miles northeast, respectively.

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. New York State Highways 96 and 96A border SEDA to the east and west, respectively. **Figure 2** presents a plan view of SEDA.

The 10,587-acre SEDA facility has been owned by the United States Government since 1941 and was operated by the Department of the Army (DOA) until 2001. From its inception in 1941 until 1995, SEDA's primary mission was the receipt, storage, maintenance, and supply of military items, including munitions and equipment. Seneca Army Depot was proposed to be included on the National Priorities List (NPL) on July 14, 1989. Once Seneca Army Depot was listed on the NPL, the Army, USEPA, and NYSDEC identified a list enumerating 57 solid waste management units (SWMUs) where historic data or information suggested, or evidence existed to support, that hazardous materials or hazardous wastes had been handled and may have possibly been released and migrated into the environment. Each of these sites was identified in the Federal Facilities Agreement (FFA) (Army, USEPA, NYSDEC, 1993) signed by the three parties, and this list subsequently expanded to include 72 sites. Activities at the SEDA are regulated by the Comprehensive Environmental Response, Compensation, and Liability Act of (CERCLA) and Resource Conservation and Recovery Act (RCRA). USEPA and NYSDEC are the approval entities for the project. The site number is listed as NY0213820830 and 8-50-006 under the USEPA and NYSDEC program, respectively.

The Depot’s mission changed in early 1995 when the Department of Defense (DoD) recommended closure of the SEDA under the Base Realignment and Closure (BRAC) process. This recommendation was approved by Congress on September 28, 1995, and the Depot was closed by July 2001.

This project is conducted by Parsons under the AFCEE Contract titled Remediation of the Seneca Army Depot Activity (FA8903-04-D-8675).

A project kickoff meeting was held on May 10, 2005. Project managers and contracting officer's representatives from Parsons, the Army, and AFCEE discussed about the project scope, schedule, and roles of various parties involved in the project.

A chemical data acquisition plan, developed in 1995 as a generic QAPP document for the Seneca Army Depot Activity, was incorporated in the Final Generic Installation Remedial Investigation/Feasibility Study (RI/FS) Workplan (Parsons, 1995) as Appendix C. This SAP, once approved, will supercede the current chemical data acquisition plan.

2.2 PROJECT SPECIFIC BACKGROUND

Background information for each specific site within the Depot will be included in the SS-WP. The SS-WP will present information of site location, site contamination history, and findings from previous investigations.

2.3 PROJECT SCOPE AND OBJECTIVES

The primary objective of the project is to conduct remedial investigation, feasibility study, and remedial action at the identified SWMUs at Seneca Army Depot. Work required includes activities such as but not limited to investigation, testing, excavation, separation, treatment, and disposal of contaminated materials. Work will be conducted in accordance with the FFA (USEPA, NYSDEC, Army, 1993), CERCLA, RCRA, National Oil and the Hazardous Substances Contingency Plan (more commonly called the National Contingency Plan, or NCP) requirements, with regulatory coordination of the NYSDEC and the USEPA Region 2.

2.4 APPLICABLE REGULATIONS/STANDARDS

Applicable or Relevant and Appropriate Requirements (ARARs) are promulgated regulatory standards or requirements and as such are legally enforceable and generally applicable and equivalent to the media or conditions at the site. In addition to ARARs, advisories, criteria, or guidance may be evaluated as "To Be Considered" (TBC) regulatory items. Comprehensive Environmental Response Compensation and Liability Action indicates that the TBC category could include advisories, criteria, or guidance that were developed by USEPA, other federal agencies, or states that may be useful in developing CERCLA remedies. The following ARARs and TBCs have been identified for the project.

Soils/Sediment

- NYSDEC Technical and Administrative Guidance Memorandum (TAGM) HWR-94-4046 (January 1994) – TBC,
- EPA Regional Preliminary Remediation Goals (PRGs) or Risk Based Concentrations (RBCs) – TBC.

Groundwater/Surface Water

- Technical Operating Guidance Series (TOGS), 1.1.1, Class GA Standards (June 1998) – ARAR
- National Recommended Water Quality Criteria – TBC
- National Primary Drinking Water Regulations - TBC

Potentially applicable ARARs and TBCs are provided in Table 1-A and 1-B for soils/sediment and groundwater/surface water, respectively.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

3.1 PROJECT ORGANIZATION

The organizations who will be directly involved in the performance of the Seneca Army Depot Activity will include the NYSDEC, USEPA Region 2, the Army, Seneca Army Depot Activity (SEDA), Air Force Center for Environmental Excellence, Parsons, and subcontractors. The organizations, key personnel from each organization, and personnel contacts are listed in the following table. A chart showing the project organization is presented in Figure 3. Project-specific responsibilities (to include any additional subcontractors) and project-specific team will be identified and discussed in detail in the SS-WP.

Organization	Position	Name & Address	Responsibility	Phone	Fax	Email
AFCEE	Contracting Officer's Representative	Lonnie Wolfe	Project Coordination	210-536-5269	(210) 536-4330	lonnie.wolfe@brooks.af.mil
Seneca Army Depot Activity	Installation Manager	Stephen Absolom Seneca Army Depot Activity, 5786 State Rte 96, P.O. Box 9 Romulus, New York 14541-0009	Project Coordination	607-869-1309	607-869-1362	stephen.m.absolom@us.army.mil
Seneca Army Depot Activity	Contracting Officer's Field Representative	Thomas Battaglia Seneca Army Depot Activity, 5786 State Rte 96, Building 125 Romulus, New York 14541-0009	Project Coordination at field, fund programming	607-869-1353		thomas.c.battaglia@nan02.usace.army.mil
Parsons	Project manager	Todd Heino 150 Federal Street, Boston, MA 02110	Overall project coordination	617-449-1405	617-946-9777	todd.heino@parsons.com
Parsons	Technical Director	John Lanier 180 Lawrence Bell Dr, Suite 104 Williamsville, NY 14221	Provide technical recommendation	(716) 633-7074 x222	1 (716) 633-7195	John.Lanier@parsons.com
Parsons	Quality Assurance Officer	James Lowerre 150 Federal Street, Boston, MA 02110	Overall QA implementation	617-449-1559	617-946-9777	jim.lowerre@parsons.com
Parsons	Field Team Leader	Tom Andrews 180 Lawrence Bell Dr., Suite 104 Williamsville, NY 14221	Sampling Operations	716-633-7074	716-633-6195	Tom.Andrews@parsons.com
Parsons	Database researcher	Eric Bishop 2701 Liberty Parkway, Suite 317 Midwest City, OK 73110-2880 Brendan Baranek-Olmstead 150 Federal Street Boston, MA 02110	Database management	405-732-9803 617-449-1404	405-732-9726 617-946-9777	Eric.Bishop@parsons.com Brendan.Baranek-Olmstead@parsons.com
Parsons	Project	Katherine Lapierre	Data	512-719-	512-719-	Katherine.L

	Chemist	8000 Centre Park Dr., Suite 200 Austin, TX 78754	Evaluation, Laboratory Coordination	6000x6806	6099	apierre@parsons.com
Parsons	Field Analyst	Ben McAllister 150 Federal Street, Boston, MA 02110	Field Analysis	617-946-1592	617-946-9777	benedict.mcallister@parsons.com
Laboratory	Lab Manager	TBD	Laboratory Analyses	TBD	TBD	TBD
NYSDEC	Project Manager	Kuldeep K. Gupta 625 Broadway Albany, NY 12233-7015	Supervision, review, and approval	518-402-9620		kxgupta@gw.dec.state.ny.us
USEPA Region 2	Project Manager	Julio F. Vazquez 290 Broadway, 18th Floor New York, NY 10007-1866	Supervision, review, and approval	212-637-4323	212-637-3256	vazquez.julio@epamail.epa.gov

3.2 ROLES AND RESPONSIBILITIES

3.2.1 USEPA and NYSDEC

For the Seneca Depot Activity, NYSDEC and USEPA are the primary regulatory agencies with responsibilities for administering the site activities. These agencies will receive copies of the SAP. All applicable communication and reports will be delivered from Parsons to SEDA for delivery to NYSDEC and USEPA. NYSDEC and USEPA are responsible for the final acceptance of all documents with authority under CERCLA and RCRA.

3.2.2 AFCEE

AFCEE is responsible for the Seneca project oversight. The overall Point of Contact for Seneca Depot activities is Mr. Lonnie Wolfe. Mr. Lonnie Wolfe or his designee will provide day-to-day liaison with the AFCEE and ensure that appropriate coordination is maintained among the different parties involved in the project. Mr. Lonnie Wolfe or his designee is responsible for programming funds, establish and maintain information repository, public involvement, and regulator and stakeholder coordination.

3.2.3 SEDA

SEDA is responsible for coordinating the activities at the Depot and has the responsibility of reviewing all supporting documents. Mr. Stephen Absolom has been designated as the installation manager for SEDA and he is responsible for ensuring that the Army's objectives are being met. Mr. Thomas Battaglia has been assigned as the contracting officer's representative. Mr. Thomas Battaglia will be responsible for day-to-day management and oversight and funding management for the Army.

3.2.4 Parsons

Parsons has been contracted for the Seneca Army Depot remediation activity and will be responsible for preparing documents and overall implementation of the remediation/investigation. Parsons team

consists of members who have extensive experience in conducting site investigation/remediation. Key personnel and their respective roles and responsibilities are discussed below.

3.2.4.1 Project Manager

Mr. Todd Heino will serve as the Parsons project manager and will have overall responsibility for implementing the project. The Boston office of Parsons is responsible for conducting the work under the contract and will be supported by other Parsons offices as needed. Mr. Todd Heino will coordinate all efforts on this project including contact with the SEDA project manager, travel for the project team, and submission of all deliverables.

3.2.4.2 Project Team

Parsons project team consists of technical personnel, including field sampling personnel, quality assurance officer, project chemist, and data users (geologists, chemists, risk assessors, engineer designers, etc.). The project team is responsible for providing all the information required by the SAP and for resolving all technical issues for the project.

Technical Director
Mr. John Lanier will perform duties of Technical Director for the SEDA activities. As Technical Director, Mr. Lanier or his designee will provide technical guidance and oversight for all field activities, and will conduct field audits and coordinate any corrective actions with the Project Manager.

Quality Assurance Officer
In accordance with the NYSDEC Technical Guidance for Site Investigation and Remediation (2002), a quality assurance officer (QAO) with the qualifications specified in the NYSDEC guidance has been assigned for the project. The quality assurance officer will review the SAP and ensure that the work be conducted in accordance with the requirements presented in the SAP and certify that the data be collected and analyzed using the appropriate procedures. The QAO, or designated team, is responsible for preparing and revising the SAP.

Field Analyst
If field analysis is planned, a qualified field analyst will be assigned to conduct the field analysis. A field analyst must have the following minimum qualifications: (1) completion of a certification course or training by an experienced analyst who has demonstrated proficiency in the method; or, (2) demonstration of the analyst's proficiency by correlation of the analyst's results with laboratory confirmation analysis.

Field Team Leader
The field team leader is an experienced person who has demonstrated proficiency in the sampling method. The field team leader is responsible for ensuring that calibration is completed daily in accordance with this procedure, that equipment and instrument inspection and maintenance is conducted, that measurements are taken to the specified accuracy, and that the requisite QA/QC samples are submitted to the laboratory.

Field Sampling Team

Field sampling team is responsible for sampling preparation, sample collection, sample storage at field, sample packaging, sample delivery, and field measurements. The team should be familiar with the SAP.

Project Chemist

The project chemist will have at least two years experience in data review and be familiar with USEPA Region 2 organic data validation requirements and the New York Analytical Services Protocol. The project chemist is responsible for data verification, data validation, and data usability evaluation for all analytical data generated for the project.

The project chemist will be responsible for communicating with the laboratory on a regular basis regarding sample shipment, receipt, and login, and all issues relating to data quality, scheduling and data packages. The project chemist will review all project and laboratory documentation related to the analytical process and will prepare data verification reports as needed.

Data Users

Technical personnel who use the collected data to perform their responsibilities (e.g., risk assessment, remedial design) will use the data for various purposes. Data users are responsible for communicating additional data needs to the project manager.

Project Health and Safety Officer

Project health and safety officer oversees the health and safety of personnel involved in the project. Project Health and Safety Officer is responsible for developing the Health and Safety Plan for the project and has the authority to initiate a work stoppage due to health and safety concerns.

3.2.5 Subcontractor

Laboratory

The laboratories selected to perform analyses for samples collected at Seneca site must be certified under the Environmental Laboratory Approval Program (ELAP), implemented by the New York State Department of Health (NYSDOH), and be capable of providing complete environmental analytical services consistent with USEPA protocols and NYSDEC Analytical Service Protocol (ASP). The laboratories should implement QA/QC procedures consistent with the NYSDEC ASP protocol, Region 2 SOPs, and this generic SAP. Prior to sample analysis, each laboratory must submit detailed information regarding the ELAP certification, laboratory project manager, and QA/QC procedures to Parsons. Parsons QA officer or project chemist will review the ELAP certification and QA/QC manual submitted by laboratories to ensure consistency with requirements by this SAP.

All analytical data will be verified prior to being released by the Laboratory. Verification will include both editorial and technical reviews. The electronic format of the data will be reviewed along with the hardcopy data package. A final review of the data package will be performed and the approved data package signed by the PM, or designee, when complete.

Other Subcontractors

Other subcontractors identified for specific project will be specified in the SS-WP. The following provides a list of potential subcontractors that may be used for the project.

General Contractors

Abscope Environmental, Inc. (Canastota, New York).

Drilling

Nothnagle Drilling (Scottsville, New York).
Northstar Drilling (Ogdensburg, New York)
SJB Services, Inc. (Hamburg, New York)

Surveys

Naybor Surveying (Alden, New York).

3.3 COMMUNICATION PATHWAYS

Communication is one of the keys to successful project. Communication pathways and modes of communication are delineated in Table 2.

The Laboratory shall communicate with Parsons PM, QA officer, or project chemist by telephone or via email as necessary throughout the process of sample scheduling, shipment, analysis and data reporting, to ensure that samples are properly processed. This shall include immediately notifying Parsons of any irregularities with samples or sample paperwork received, noting discrepancies between paperwork and verbal orders placed by Parsons authorized personnel, problems encountered in sample analyses that could affect data quality or schedule, and any laboratory conditions that may impact the timeliness of analyses or data reporting. In particular, the Laboratory shall notify Parsons in advance regarding any data that could potentially be late and shall specify an estimated delivery date.

The field team leader shall communicate with the project manager or QA officer by telephone as necessary throughout the sampling event to ensure that samples are properly collected and delivered.

4.0 DATA QUALITY OBJECTIVES

Data Quality Objectives (DQOs) define the type, quantity, and quality of data that are needed to answer specific environmental questions and support environmental decisions. The development of DQOs for a specific site and measurement takes into account project needs, data uses and types and needs, and data collection. These factors determine whether the quality and quantity of data are adequate for its end use. DQOs are implemented so the data are legally and scientifically defensible. This section presents the general process of DQO development and factors that will affect DQO development (Sections 4.1 and 4.2). Site-specific DQOs will be defined in the SS-WP. As part of the DQO development, data performance criteria need to be determined. Section 4.3 presents indicators that will be used to represent data quality and their performance requirement criteria. Section 4.5 and Section 4.6 describe quality control activities and quality control checks for the project, which will be conduced to ensure the data quality.

4.1 DATA QUALITY OBJECTIVE DEVELOPMENT PROCESS

Development of data quality objectives will be conducted in accordance with the USEPA (2000) Guidance for the Data Quality Objectives Process Technical Project Planning, the USEPA (2004) Uniform Federal Policy for Quality Assurance Project Plans, and the NYSDEC (2001) Development and Review of Site Analytical Plans.

The following elements will be incorporated into the DQO development in accordance with the NYSDEC guidance and the sections corresponding to the elements are specified:

- definition of data types and data uses (Section 4.2),
- specification of data performance criteria (Section 4.3),
- discussion of implementation mechanisms of sampling for routine, baseline and expanded parameters (Sections 4.4 and 4.5)
- presentation of action levels or applicable standards (Section 2.4)

A Systematic Planning Process (SPP) described in the USEPA (2004) guidance or the Data Quality Objectives Process (DQOP) discussed in the USEPA (2000) guidance will be used to identify site-specific DQOs based on the specific site information.

The SPP process, presented in Figure 13 of the USEPA (2004) guidance, is based on the scientific method and includes concepts such as objectivity of approach and acceptability of results. It uses a common sense graded approach to ensure that the level of detail in planning is commensurate with the importance and intended purpose of the work and the use of available resources. This framework promotes communication between all organizations and individuals involved in an environmental project.

When critical environmental decisions need to be made (e.g., final decision-making or compliance with a standard), the USEPA (2000) defined DQOP will be followed. The DQOP requires statistical expertise to define the amount of error acceptable when making an environmental decision and includes the following seven steps:

- Step 1: State the problem

- Step 2: Identify the decision
- Step 3: Identify the inputs to the decision
- Step 4: Define the boundaries of the study
- Step 5: Develop a decision rule
- Step 6: Specify tolerable limits on decision errors
- Step 7: Optimize the design for obtaining data

The DQO process is iterative, i.e., the seven-step process should be repeated, as needed, based on newly acquired data and/or information.

DQO for a specific project will be presented in a site-specific work plan. In general, the DQO will be developed using the SPP or DQOP for the Data Quality Objectives Process. Most projects under the contract will be judgmental-based and therefore SPP, a less iterative process, is normally used to develop the project's data quality criteria.

4.2 DATA TYPES AND DATA USES

DQOs are based on the premise that different data types or different data uses require different levels of data quality. This section provides information on potential data type (Section 4.2.1) and data uses (Section 4.2.2) for the project.

4.2.1 Data Types

Two types of data will be produced for the Seneca Depot Activity. Screening data are data generated by rapid methods of analysis with less rigorous sample preparation, calibration and/or QC requirements. Physical test methods (e.g., dissolved oxygen measurements, temperature, pH, moisture content, turbidity, conductance, etc.) have been designated by definition as screening methods. Screening data are to be used for screening purposes. All screening methods are presented in Table 3.

Definitive data are analytical data that are suitable for final decision-making. All definitive data will be generated using rigorous analytical methods such as approved EPA SW-846 reference methods. All definitive methods are presented in Table 4. Tables 5-A and 5-B present sample containers, preservatives, and holding times for soils/sediment and aqueous samples, respectively. Table 6-A through Table 6-G present target analyte list for various analytical methods and Table 7-A through Table 7-F present quality control requirements for various analytical methods.

In addition to the above referenced two types of data, nonmeasurement data acquisition may be required for each project. The data that may be required include:

- Climate,

- Geology and soils,
- Hydrogeology,
- Local relevant habitats, and
- Threatened and endangered species.

4.2.2 Data Uses

Data produced under the project will be used by various users for a variety of purposes, such as determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, determining when remedial actions are complete, and determining compliance with regulatory permit limits and environmental standards. The data may be used in all stages in the investigation of a SWMU, including site inspections, remedial investigations/feasibility studies, remedial design, treatability studies, and removal actions. In addition, the data may be used in enforcement/litigation activities.

4.3 DATA QUALITY INDICATORS

This section presents a brief introduction of the data quality indicators (DQIs) including precision, accuracy, representativeness, completeness, comparability, sensitivity, and defensivity. The quantitation method for each indicator is discussed in this section. The measurement performance criteria for each indicator identified for the project is presented in Table 7-A through Table 10-C.

4.3.1 Precision

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed conditions. Assessing precision measures the random error component of the data collection process. Precision is determined by measuring the agreement among individual measurements of the same property, under similar conditions, and is calculated as an absolute value. The degree of agreement, expressed as the relative percent difference (RPD), is calculated using the formula below.

$$\text{RPD} = \frac{(V_1 - V_2)}{\frac{(V_1 + V_2)}{2}} \times 100$$

where: V1 = value 1; V2 = value 2

Analytical precision can be assessed by analyzing matrix spike/matrix spike duplicate pairs, laboratory control spike/laboratory control spike duplicate pairs, and laboratory analytical duplicate samples. Field precision is assessed by measurement of field duplicate samples. The objective for precision is to be within the established control limits for the methods. A note will be provided if

RPD is not calculated due to missing data values, "less than" or "greater than" values, or other reasons. The control limits for precision are presented in Table 7-A through Table 7-F, Table 10-A through Table 10-F, and Table 12 and any exceedance of the values listed in the table will trigger corrective actions as presented in Section 11.

4.3.2 Accuracy/Bias

Accuracy is the degree of agreement of a measurement with an accepted reference or true value; bias indicates the systematic or persistent distortion of a measurement process that causes errors in one direction. The terms accuracy and bias are used interchangeably in this document. Accuracy measures the bias or systematic error of the entire data collection process. Sources of these errors include the sampling process, field and laboratory contamination, sample preservation and handling, sample matrix interferences, sample preparation methods, and calibration and analytical procedures. To determine accuracy, a reference material of known concentration is analyzed or a sample which has been spiked with a known concentration is reanalyzed. Accuracy is expressed as a percent recovery and is calculated using the following formula:

$$\%\ \text{Recovery} = 100 \times \frac{\text{measured value}}{\text{true value}}$$

Recoveries are assessed to determine method efficiency and matrix interference effects. Analytical accuracy is measured by the analysis of calibration checks, system blanks, quality control samples, surrogate spikes, matrix spikes, and other checks required by the selected analytical methods. Sampling accuracy is assessed by evaluating the results of field and trip blanks. Sampling accuracy is also maintained by frequent and thorough review of field procedures. The objective of accuracy is to meet the established control limits for the methods. A note will be provided if % recovery is not calculated due to missing data values, "less than" or "greater than" values, or other reasons. The control limits for precision are presented in Table 7-A through Table 7-F and Table 10-A through Table 10-C and any exceedance of the values listed in the table will trigger corrective action requirements as presented in Section 11.

4.3.3 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is achieved through proper development of the field sampling program. The sampling program must be designed so that the samples collected are as representative as possible of the medium being sampled and that a sufficient number of samples will be collected.

4.3.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Data are complete and valid if they meet all acceptance criteria including accuracy, precision, and any other criteria specified by the particular analytical method being used. Data with minor exceedances in accuracy and precision my also be considered usable.

Field completeness will be estimated as the percentage of all planned samples that were actually collected and analyzed. The calculation is as follows:

$$\% FC = (A/P) \times 100$$

where,

%FC = Field Percent Completeness;
A = Actual number of samples collected; and
P = Number of planned samples to collect.

Laboratory completeness will be estimated as the percentage of all usable measurements and calculated as follows:

$$\%C = (U/T) \times 100$$

where:

%C = Percent completeness;
U = Number of measurements judged usable; and
T = Total number of measurements.

The objective is to generate a sufficient database with which to make informed decisions. To help meet the completeness objective, every effort must be made to avoid sample loss through accidents or inadvertence. The required completeness for a project will be defined by the SS-WP.

4.3.5 Comparability

Comparability expresses the confidence with which one data set can be compared to another. Comparability must be considered in designing the sampling program and the objective will be met by using standard methods for sampling and analyses specified in this report and by following techniques and methods set forth in the SS-WP.

Whenever definitive analysis is performed to confirm screening results, comparability criteria must be established and documented in the SS-WP prior to data collection. Comparability criteria must be determined for each matrix, analytical group (and analyte, if applicable), and concentration level.

4.3.6 Sensitivity and Quantitation limits

Sensitivity is the ability of the method or instrument to detect the target analytes at the level of interest. Method and instrument sensitivity is measured by developing Method Detection Limits (MDLs) for each analyte of interest. The MDL is a statistically derived value that represents the lowest concentration of an analyte that can be detected with 99 percent confidence that the analyte concentration is greater than zero. And MDL study is performed for each analyte, instrument and matrix and represents the lowest concentration detectable under those conditions.

The quantitation limit (QL) is the minimum concentration of an analyte that can be routinely identified and quantified by a laboratory. The QL is usually three to five times the MDL. For multipoint calibrations, the lowest point of the calibration curve should be at or below the QL. For one-point calibrations, the laboratory should analyze a check standard that contains all target analytes at or below the QL as proof that the analyte can be quantitated at that level.

4.3.7 Defensibility

Data defensibility is defined as data that are both relevant and reliable. This generic SAP was designed to improve data defensibility for the project. A few key elements that will ensure data defensibility are:

- Appropriate documentation, including Chain of Custody forms, project records and analytical traceability
- Using appropriate and approved analytical methodology
- Using NYSDOH ELAP-certified laboratories
- Appropriate sampling design, sample collection, sample handling, and sample storage
- Data validation
- Audits

4.4 METHOD DETECTION LIMITS, REPORTING LIMITS, AND INSTRUMENT CALIBRATION REQUIREMENTS

This section describes the terminology, procedures, and laboratory-established values for method detection and reporting limits. Definitions for method detection limits, and reporting limits are described below.

4.4.1 Method Detection Limit (MDL)

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence when the analyte concentration is greater than zero. The MDL is lower than the concentration at which the laboratory can quantitatively report. Laboratories determine their "best case" sensitivity for analytical methods by performing MDL studies. The MDL determinations are performed annually by following the USEPA SW846 methods. MDLs generated by the chosen laboratory, will be attached as an addendum to the SS-WP. A laboratory certified by the NYSDOH ELAP laboratory certification program will be selected for the Seneca Army Depot Activity.

4.4.2 Method Detection Limit Verification

An MDL verification check shall be performed on each instrument immediately following an MDL study and can be performed quarterly in place of the annual (every 12 months) MDL study. However, this may not substitute for the initial MDL determination. The MDL check sample shall be spiked at approximately 2 times the current reported MDL and taken through all preparatory and analytical steps. The MDL is verified if the laboratory can reliably detect and identify all analytes in the check sample by the method-specified criteria. If the method has no confirmation criteria, the check sample must produce a signal that is at least 3 times the instrument's noise level. If the MDL is not verified, spike at successively higher concentrations until the verification criteria are met, and use the first successful concentration as the reported MDL.

4.4.3 Sample Quantitation Limit (SQL)

Frequently, quantitation limits (QLs) for specific samples are adjusted for dilutions, changes to sample volume/size and extract/digestate volumes, percentage of solids, and cleanup procedures. These QLs are referred to as sample quantitation limits (SQLs).

4.4.4 Action Limit (AL)

The action limit (AL) for a target analyte is the numerical value the decision-maker uses as the basis for choosing one of the alternate actions. It may be a regulatory threshold such as maximum contaminant levels (MCLs), a risk-based concentration level, a reference-based standard, or a technological limitation. The action limits identified for selected potential contaminants of concern for the project are listed in Tables 1-A and 1-B.

SQLs must be less than the action limits for project quality objectives to be definitively met. Sample results that are reported to SQLs that are higher than the action limits cannot be used to determine whether the action limit has been exceeded. Thus, environmental decision-making may be adversely affected by the failure to meet SQLs.

Because of uncertainty at the quantitation limit, project SQLs should be no greater than one-third of the action limit and ideally one-tenth of the action limit.

4.4.5 Reporting Limit (RL)

The laboratory participating in any project under this contract shall compare the results of the MDL demonstrations to the reporting limit (RL) for each analyte. Laboratory RLs should be at least 3 times the achievable laboratory MDL and ideally 10 times the achievable laboratory MDL. The laboratory shall also verify RL by including a standard at or below the RL as the lowest point on the calibration curve. All results shall be reported at or above the MDL values; however, for those results falling between the MDL and the RL, an F-flag shall be applied to indicate the analyte was detected, but the concentration is an estimation. No results shall be reported below the MDL. The reporting limit must be at or below the project quantitation limits presented in Tables 1-A and 1-B or SS-WP unless otherwise approved by the project manager.

4.4.6 Contract Required Quantitation Limit/Contract Required Detection Limit

Contract Required Quantitation Limit (CRQL) is the minimum level of reliable quantitation acceptable under the USEPA Contract Laboratory Program (CLP). The organic contract Statement of Work (SOW) for the Contract Laboratory Program gives CRQLs, and they are used for reporting limits (after adjustment for %moisture and dilution). The CLP CRQLs are arbitrarily set at the concentration of the lowest non-zero standard in the calibration curve. Organic analytes that are positively identified below the CLP CRQL are reported as present, but at an estimated concentration (with a "J" flag). The laboratory RL should be reported below the CRQL under the CLP program.

Contract Required Detection Limit (CRDL) is the minimum level of detection acceptable under the contract Statement of Work (SOW). The inorganic SOW for the Contract Laboratory Program gives CRDLs, but laboratory-derived IDLs (adjusted for sample size, dilution and moisture) are used for reporting limits. The CLP CRDLs are based on typical instrument capabilities and should be attained

by the laboratory. Inorganic analytes reported at a concentration above the laboratory's IDL but below the CLP CRDL are flagged with a "B".

4.4.7 Instrument Calibration

Measuring and testing instrument shall have an initial calibration and shall be recalibrated/verified at scheduled intervals against certified standards that have known and valid traceability to recognized national standards. Calibration intervals for each item shall be, at a minimum, in accordance with manufacturer's recommendations as defined in the instrument manual, the analytical method, the NYSDEC ASP, and the project specific QA requirements.

Calibration standards shall be maintained and used in an environment with temperature, humidity, and cleanliness controls that are compatible with the accuracy and operating characteristics of the standards. An inspection will be made during the instrument calibration to evaluate the physical condition of the instrument. The purpose of the inspection is to detect any abnormal wear or damage that may affect the operation of the instrument before the next calibration. Instrument found to be out of calibration or in need of maintenance or repair will be identified and removed from service.

The laboratory QA Officer shall be notified if the instrument is found to be out of tolerance during inspection and calibration. The corrective actions to be taken include evaluating the validity of previous inspection or test results; evaluating the acceptability of the items inspected or tested since the last calibration check; and repeating the original inspections or tests using calibrated instrument when it is necessary to establish the acceptability of previous inspections or tests. Specifics regarding QC checks and verification of equipment stability are presented in Table 7-A through 7-F for laboratory instrument and Table 11 for field instrument.

All measuring and testing instrument use shall have current documentation of the calibration status and calibration expiration date. Instrument history records for measurement and test equipment shall be used to indicate calibration status and conditions, corrections to be applied, results of in-service checks, and repair history. This will provide a basis for establishing calibration frequencies and for remedial action if the instrument is found out of calibration.

4.4.7.1 Laboratory Instrument Calibration

Analytical instruments shall be calibrated in accordance with the analytical methods. All target analytes reported shall be present in the initial and continuing calibrations, and these calibrations shall meet the acceptance criteria specified in Table 7-A through Table 7-F. All results reported shall be within the calibration range. Results outside the calibration range are unsuitable for quantitative work and only give an estimate of the true concentration. For SW6010 and SW6020, results shall be within the working linear range determined by linear range studies performed in accordance with the method and NYSDEC ASP. Records of standard preparation and instrument calibration shall be maintained.

Records shall unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards shall be traceable to standard materials. Instrument calibration shall be checked using all target analytes identified in the project-specific requirements and surrogates. If no project-specific analytes are identified, the analytes listed in Table 6-A through Table 6-G shall serve as the default analytes for the method.

This applies equally to multicomponent analytes (e.g., PCBs). All calibration criteria shall satisfy NYSDCE ASP at a minimum. The initial calibration (ICAL) must be verified by a second source standard. Multipoint calibrations shall contain the minimum number of calibration points specified in Tables 7-A with all points used for the calibration being contiguous. If more than the minimum number of standards is analyzed for the initial calibration, all of the standards analyzed shall be included in the initial calibration. The only exception to this rule is a standard at either end of the calibration curve can be dropped from the calibration, providing the requirement for the minimum number of standards is met.

Acceptance criteria for the calibration are presented in Table 7-A through Table 7-F. Analyte concentrations are determined with either calibration curves or response factors (RFs). For gas chromatography (GC) and GC/mass spectrometry (GC/MS) methods, when using RFs to determine analyte concentrations, the average RF from the initial calibration shall be used. The continuing calibration shall not be used to update the RFs from the initial calibration. The continuing calibration verification (CCV) cannot be used as the laboratory control sample (LCS), except for methods that do not involve sample preparation (e.g., volatile organic analysis). A CCV is to be performed daily before sample analysis (unless an initial calibration and second-source standard verification is performed immediately before sample analysis) and as required by the applicable method and the SAP (Table 7-A through 7-F gives the appropriate frequencies.). In addition, the concentration used for the CCV sample shall be at or below the middle of the calibration curve. Finally, the lowest standard used must be at or below the RL for each analyte in the method.

If calibration acceptance criteria are not met, corrective action will be implemented and recalibration conducted, and the laboratory will reanalyze all samples since last successful calibration verification.

4.4.7.2 Field Instrument Calibration

The frequency of calibration for field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate, but daily as a minimum.
To ensure comparability between sample data of similar samples and sample conditions, standard solutions and material traceable to the National Institute of Standards and Technology or USEPA-published standards/protocols will be used to calibrate the field instruments.

Table 11 summarizes requirement for field equipment calibration, maintenance, testing, and inspection.

4.5 QUALITY CONTROL ACTIVITIES

QC elements relevant to screening data are presented in Section 6.0. This section presents QC requirements relevant to analysis of environmental samples that shall be followed during all analytical activities for fixed-base, mobile, and field laboratories producing definitive data. The purpose of these QC activities is to produce data of known quality that satisfy the project quality objectives (PQOs). These activities provide a mechanism for ongoing control and evaluation of data quality measurements through the use of QC materials.

Laboratory quality control samples (e.g., method blanks and Laboratory Control Spike samples) shall be included in each preparation batch with the field samples. A project analytical batch (PAB) is a

group of samples (not exceeding 20 environmental samples plus associated laboratory QC samples) that are similar in composition (matrix) which are extracted or digested at the same time and with the same lot of reagents and analyzed together as a group. The term "PAB" also extends to cover samples that do not need separate extraction or digestion (e.g., volatile analyses by purge and trap). The identity of each PAB shall be unambiguously reported with the analyses so that a reviewer can identify the QC samples and the associated environmental samples. All references to the analytical batch in the following sections and tables in this SAP refer to the PAB.

The following sections summarize quality control activities for the Seneca Army Depot Activity including laboratory selection requirement and QC sample requirement.

4.5.1 Laboratory Certification, Qualification, and Selection

To be selected for project chemical analysis, the laboratory should be certified by the NYSDOH ELAP program. The following four laboratories have been identified as potential laboratories for the project.

1) Severn Trent Laboratories, Inc.
   (STL Pittsburgh)
   301 Alpha Drive
   Pittsburgh, PA 15238
   Contact: Mr. David Miller

2) Columbia Analytical Services
   1 Mustard St., Suite 250
   Rochester, NY
   Contact: Mark Wilson

3) General Engineering Laboratories
   PO Box 30712
   2040 Savage Road
   Charleston, SC 29417
   Contact: Nancy Mattern

4) Severn Trent Laboratories, Inc.
   (STL Buffalo)
   10 Hazelwood Drive, Suite 106
   Amherst, NY 14228
   Contact: Tony Bogolin

For each specific project, the project team will identify appropriate laboratory that conforms to the requirements presented in this SAP. In brief, the laboratory should follow the requirements presented below:

- For the analysis of any aqueous samples for a parameter or category of parameters for which laboratory certification exists pursuant to NYSDOH ELAP Certification, the laboratory should be certified for that specific parameter or category of parameters pursuant to NYSDOH ELAP Certification;

- For the analysis of non-aqueous samples using specific analytical methods contained in the EPA Publication SW-846, "Test Methods for Evaluating Solid Waste", third edition, update IIF, January 1995, as amended and supplemented, for a parameter or category of parameters for which certification exists pursuant to NYSDOH ELAP Certification, the laboratory will be certified for that specific parameter or category of parameters pursuant to NYSDOH ELAP Certification or, at a minimum, have obtained temporary approval to analyze regulatory samples pursuant to NYSDOH ELAP Certification;
- The reporting limits for chemicals of potential concern should be within the limits specified in the SAP or SS-WP;
- The laboratory should follow the QA/QC procedures described in the NYSDEC ASP;
- The laboratory should report the analytical results consistent with the NYSDEC ASP requirement and those specified in the SAP (Section 8.2.1);
- The laboratory shall provide an electronic data deliverable (EDD) in the Environmental Restoration Program Information Management System (ERPIMS) format as specified in the AFCEE Guidance for Contract Deliverables, Appendix C - Quality Assurance Project Plan (QAPP, Version 4.0, 2005).

The laboratory identified for the project will be specified in the site-specific work plan.

4.5.2 Laboratory Control Sample

The Laboratory Control Sample (LCS) is a blank matrix(contaminant-free water or an inert solid such as glass beads or Teflon chips) that is spiked with a known concentration of all target analytes. Each analyte in the LCS shall be spiked at a level less than or equal to the midpoint of the calibration curve (The midpoint is defined as the median point in the curve, not the middle of the range).

The LCS shall be carried through the complete sample preparation and analysis procedure. At least one LCS shall be included in every PAB. If more than one LCS is analyzed in a single PAB, results from all LCS samples shall be reported. The failure of any analyte in the LCS shall require appropriate corrective action, including possible qualification of the failed analyte in all associated samples.

4.5.2.1 LCS Control Limits

The LCS control limits are presented in Table 7-A through Table 7-F and Table 9-A through Table 9-F. The limits are based on those specified in the NYSDEC ASP and the USEPA Region 2 SOPs. The laboratory may use in-house LCS control limits. However, those limits must be within the LCS control limits listed in the tables, if applicable. The performance of the LCS is evaluated against the control limits. When an analyte in the LCS exceeds the upper or lower control limit and no corrective action is performed or the corrective action is ineffective, the appropriate flag, consistent with the USEPA Region 2 SOPs, shall be applied to all affected results. Once an LCS has failed (as specified in Table 7-A through Table 7-F), corrective action is required.

4.5.2.2 LCS Corrective Action

If a sample fails based on the criteria presented in Table 7-A through Table 7-F, corrective action is required. The corrective action requirement applies to all analytes that exceeded the LCS control limits, even if one specific analyte's exceedance was not the trigger of LCS failure.

If an LCS fails, an attempt must be made to determine the source of error and find a solution. All the findings and corrective action should be documented. If a systematic problem is found, the problem should be resolved and system control reestablished. Following the reestablishment of control, all samples in the PAB shall be re-prepared and reanalyzed for the out-of-control analyte(s). The corrective action applied shall be based on professional judgment in the review of other QC measures (i.e., surrogates). If an analyte falls outside the LCS control limits a second time or if there is not sufficient sample material available to be reanalyzed, then all the results in the PAB for that analyte will be flagged in accordance with the USEPA Region 2 SOPs. The recoveries of those analytes subject to corrective action must be documented in the case narrative, whether flagging is needed or not.

4.5.3 Matrix Spike/Matrix Spike Duplicate

A matrix spike (MS) and matrix spike duplicate (MSD) is an aliquot of sample spiked with known concentrations of all target analytes. The spiking occurs prior to sample preparation and analysis. Each analyte in the MS and MSD shall be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte. Only project samples shall be used for spiking. The MS/MSD shall be designated on the chain of custody. Matrix spikes (MS) and matrix spike duplicates (MSDs) are treated as environmental samples.

The MS/MSD pair is used to document potential matrix effects associated with a site. Parsons project managers must select the samples for MS/MSD analysis. The sample replicates will be collected in the field and will be used by the laboratory to prepare and analyze the appropriate MS/MSDs. Only one soil sample container may be necessary for the parent sample, the MS sample, and the MSD sample with the exception of volatile organic analysis (VOA).

A site-specific MS/MSD should be specified for each media (e.g., any different soil, water, or sediment) at each site during each sampling event. Project managers should designate the MS/MSD and determine whether they are site specific based on the project requirements. A minimum of one MS and one MSD shall be designated by the project manager for each site and included for every 20 field samples (i.e., collect up to 20 field samples followed by two additional samples designated as MS and MSD). More than one MS/MSD pair may be submitted as part of the sample group of environmental samples; however, project managers must coordinate with the laboratory providing analytical services for most cost effective sampling. Based on the projects size and duration, it is possible that not every sample delivery group will include an MS/MSD pair. This is acceptable provided the overall project requirements are met.

The performance of the MS and MSD is evaluated against the QC acceptance limits given in Table 7-A through 7-F and Table 10-A through 10-C. If either the MS or the MSD is outside the QC acceptance limits, the data shall be evaluated to determine whether there is a matrix effect or analytical error and whether the analytes in all related samples shall be qualified according to the USEPA Region 2 SOPs. The laboratory should communicate potential matrix difficulties to Parsons so an evaluation can be made with respect to the PQOs.

4.5.4 Surrogates

Surrogates are compounds similar to the target analyte(s) in chemical composition and behavior in the analytical process but not normally found in environmental samples.

Surrogates are used to evaluate accuracy, method performance, and extraction efficiency. Surrogates shall be added to all environmental samples, controls, and blanks, in accordance with the method requirements.

Whenever a surrogate recovery is below 10%, corrective action must be performed. If systematic problems are found, the problems should be resolved and system control reestablished. After the reestablishment of control, the affected sample(s) should be re-prepared and reanalyzed. If corrective actions are not performed or are ineffective, or if sufficient sample volume is not available for reanalysis, the appropriate flag, consistent with the USEPA Region 2 SOPs, shall be applied to the sample results. Table 8 presents performance criteria for surrogate recovery.

4.5.5 Internal Standards

Internal standards (ISs) are known amounts of standards added to a portion of a sample or sample extract and carried through the entire determination procedure. They are used as a reference for calibration and for controlling the precision and bias of the analytical method.

ISs shall be added to all environmental samples, controls, and blanks, in accordance with the method requirements.

When the IS results are outside of the acceptance limits, corrective actions shall be performed. If systematic problems are found, the problems should be resolved and system control reestablished. After the reestablishment of control, the affected sample(s) should be re-prepared and reanalyzed. If corrective actions are not performed or are ineffective, or if sufficient sample volume is not available for reanalysis, the appropriate flag, in accordance with the USEPA Region 2 SOPs, shall be applied to the sample results.

4.5.6 Retention Time Windows

Retention time (RT) windows are used in GC, ion chromatography (IC) and high performance liquid chromatography (HPLC) analysis for qualitative identification of analytes. They are calculated from replicate analyses of a standard performed on multiple days. The procedure and calculation method are given in SW-846, Method 8000C. The center of RT window is established for each analyte and surrogate using the RT of the mid-point standard of the initial calibration. For non-MS methods, the retention times for each analyte are updated daily using the absolute RTs from the calibration verification performed at the beginning of each PAB.

If a significant shift in RTs is observed, the analyses should be halted and the instrumentation should be inspected to identify the cause of the shift. After any systematic problems have been resolved and system control has been reestablished, reanalyze all samples run after the shift occurred. If corrective actions are not performed, the appropriate flag, in accordance with the Region 2 SOPs, shall be applied to the sample results.

4.5.7 Interference Check Samples

Interference check samples (ICSs) are used in inductively coupled plasma-atomic emission spectra (ICP-AES) and Inductively Coupled Plasma-Mass Spectra (ICP-MS) analyses only and contain known concentrations of both interfering and analyte elements.

The ICSs are used to verify background and interelement correction factors.

The ICSs are run at the beginning of each run sequence for SW6010B and SW6020B.

When the interference check sample results are outside of the acceptance limits given in Table 7-C and Table 7-D, corrective action shall be performed. After the system problems have been resolved and system control has been reestablished, reanalyze the ICSs. If the ICS results are acceptable, reanalyze all affected samples. If corrective action is not performed or the corrective action was ineffective, the appropriate flag, in accordance with the USEPA Region 2 SOPs, shall be applied to all affected results.

4.5.8 Method Blank

A method blank is an analyte-free matrix carried through the complete sample preparation and analytical procedure. The method blank is used to assess possible contamination resulting from the preparation or analytical process. A method blank shall be included in every PAB.

The presence of analytes in a method blank at concentrations greater than the MDL indicates the need for further assessment of the data. The source of contamination should be investigated and measures must be taken to correct, minimize, or eliminate the problem if the concentration exceeds the RL or CRQL/CRDL. For common laboratory contaminants (e.g., methylene chloride, acetone, phthalates), the concentration found in the method blank must not exceed the limits specified in Table 7-A through 7-F. No analytical data shall be corrected for the presence of analytes in blanks. When an analyte is detected in the method blank and in the associated samples and corrective actions are not performed or are ineffective, the appropriate flag, as described in the USEPA Region 2 SOPs, shall be applied to the sample results.

4.5.9 Equipment Blank

An equipment blank is a sample of American Society for Testing and Materials (ASTM) Type II reagent grade water poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis. These may also be called rinse blanks or rinsate blanks.

Equipment blanks are used to assess the effectiveness of equipment decontamination procedures.

Equipment blanks shall be collected immediately after the equipment has been decontaminated and included for each sampling event as appropriate. The equipment blank samples shall be analyzed for all parameters requested for the environmental samples collected at the site.

When an analyte is detected in the equipment blank, the appropriate flag, as described in USEPA Region 2 SOPs, shall be applied to all sample results from samples collected with the affected equipment.

4.5.10 Trip Blank

The trip blank consists of a volatile organic compound (VOC) sample vial filled in the laboratory with ASTM Type II reagent grade or organic-free water, transported to the sampling site, handled like an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are only submitted when samples are collected and analyzed for VOC analytes.

Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. Each cooler sent to the laboratory which contains samples for analysis of VOCs shall contain one trip blank.

When an analyte is detected in the trip blank and in the associated samples, the appropriate flag, as described in the USEPA Region 2 SOPs, shall be applied to all sample results from samples in the cooler with the affected trip blank.

4.5.11 Field Duplicate (Replicate) Samples

Field duplicates are two (or more) field samples taken at the same time in the same location. They are intended to represent the same population and are taken through all steps of the analytical procedure in an identical manner. These samples are used to assess precision of the entire data collection activity, including sampling, analysis, and site heterogeneity.

Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and are treated in an identical manner during storage, transportation, and analysis. The samples may be either collocated samples or subsamples (replicates) of a single sample collection. Examples of collocated samples include ambient air monitoring samples, surface water grab samples, and side-by-side soil core samples, while subsamples may be taken from one soil boring or sediment core. The sample containers are assigned a unique identification number in the field. Specific locations should be designated for collection of field duplicate samples prior to the beginning of sample collection.

A minimum of one duplicate or replicate sample shall be included for every 20 field samples. Precision acceptance criteria are given in Table 12.

4.5.12 Laboratory Duplicate Samples

Laboratory duplicate samples, also known as analytical duplicates, demonstrate the precision of the analytical process within the laboratory. A minimum of one analytical duplicate sample shall be performed for every 20 field samples. Acceptance criteria are given in Table 12.

4.6 QUALITY CONTROL CHECKS

This section summarizes quality control checks including sample holding time compliance check, quantitation confirmation for samples analyzed using GC or HPLC, standard material check, and supplies and consumables check.

4.6.1 Holding Time Compliance

To maximize representativeness of sample results, all samples will be extracted and/or analyzed within the holding times specified in each method. Tables 5-A and 5-B present the maximum holding times allowed for each method. Extraction or analysis performed after the expiration of the holding time will result in the qualification of the results during the data validation procedures.

Any samples that exceed project required holding time for extraction or analysis may be resampled and resubmitted for analysis.

It should be noted that the NY ASP requires holding times to be calculated from the verified time of sample receipt (VTSR) and not from the sample collection date and time. Tables 5-A and 5-B list both technical holding time requirement and NY ASP holding time requirement.

4.6.2 Quantitation Confirmation

Quantitative confirmation of results at or above the RL for samples analyzed by GC or HPLC shall be required, unless otherwise specified in the SS-WP, and shall be completed within the method-required holding times. If holding times are exceeded and the analyses are performed, the results shall be flagged according to the USEPA Region 2 SOPs. For GC methods, a second column is used for confirmation. For HPLC methods, a second column or a different detector will be used. Unless otherwise specified or overlapping peaks are causing erroneously high results, the lower of the two confirmed results shall be reported as the primary result. The column used for both the primary and confirmation results shall be indicated on the sample reports. The associated calibration and QC results (including method blank, LCS, MS/MSD, surrogates and internal standards) shall be submitted for both columns so that sample results can be appropriately evaluated.

4.6.3 Standard Materials

Standard materials, including second source materials, used in calibration and to prepare samples shall be traceable to National Institute Standards and Technology (NIST), USEPA, American Association of Laboratory Accreditation (A2LA) or other equivalent AFCEE approved source, if available. If an NIST, USEPA, or A2LA standard material is not available, the standard material proposed for use shall be included in an addendum to the laboratory QA manual submitted to Parsons before the analyses. The standard materials shall be current, and the following expiration policy shall be followed: The expiration dates for standards shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first. Expiration dates for laboratory prepared stock and diluted standards shall be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals shall be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials shall be either revalidated prior to use or discarded. Revalidation may be performed through assignment of a true value and error window statistically derived from replicate

analyses of the material as compared to an unexpired standard. The laboratory shall label standard and QC materials with expiration dates.

A second source standard is used to independently confirm the initial calibration. A second source standard is a standard purchased from a vendor different from that supplying the material used in the initial calibration. The second source material can be used for the continuing calibration standards and/or for the LCS. Two different lot numbers from the same vendor do not normally constitute a second source. However, when a project requires analyses for which there is not a separate vendor source available, the use of different lot numbers from the same vendor will be acceptable to verify calibration.

4.6.4 Supplies and Consumables

The laboratory shall inspect supplies and consumables prior to their use in analysis. The materials description in the methods of analysis shall be used as a guideline for establishing the acceptance criteria for these materials. Purity of reagents shall be monitored and documented. An inventory and storage system for these materials shall assure use before manufacturers' expiration dates and storage under safe and chemically compatible conditions. As part of the laboratory's maintenance program, service contracts are held on sufficient supplies. SOP's for routine maintenance of supplies and consumables shall be submitted for each laboratory performing analytical services as part of this project. Consistent with the Uniform Federal Policy for Quality Assurance Project Plans, the documentation should include the following:

- Supplies that will be used in the performance of analytical work
- All vendors for supplies and reagents
- Specifications for all supplies and reagents that could affect data quality (such as level of contamination, pesticide versus reagent-grade). Procedures that will be used to ensure supply cleanliness and reagent purity (such as recording reagent lot numbers)
- Procedures for measuring supply cleanliness
- Corrective action procedures for preventing the use of unacceptable supplies

The laboratory shall purchase or prepare sample containers in accordance with the specifications in the NYSDEC ASP (Exhibit I) and the SAP (Table 5-A and Table 5-B), unless specifically directed to do otherwise by Parsons. The individuals responsible for checking supplies and implementing corrective actions will be identified by the laboratory. Laboratory QA manuals, which include supplies and consumables inspection SOP, will be reviewed by Parsons project chemist before the analysis starts.

Supplies and consumables for field activities will be inspected by field team leader. Table 11 presents inspection requirement and Table 13 provides a critical supplies and consumable tracking log.

5 SAMPLING PROCEDURES

5.1 FIELD SAMPLING PROCEDURES

Field sampling procedures including field sample collection SOPs, field sample storage, and sample handling and custody are presented in Section 16.

5.2 SAMPLE COLLECTION DOCUMENTATION

Documentation for sample collection includes sample container identification, field notes recording any observation during the sample collection, and the chain-of-custody discussed in detail in the following section.

The sample label requirement is discussed in detail in Section 16. The information on the label will be preserved by covering the label with clear tape to minimize water damage during transit. Requirement for other field documentation (e.g., field logbooks and field data collection forms) is presented in Section 16.

5.3 SAMPLE HANDLING AND CUSTODY

A sample is physical evidence collected from a facility or from the environment. Controlling evidence is an essential part of the hazardous waste investigation effort. To accomplish this, proper sample handling and custody procedure should be followed.

5.3.1 Sample Identification

To assure traceability of the samples, samples should be properly labeled in the field with assigned sample identification (ID). The Laboratory shall have a specified method for maintaining identification of samples throughout the Laboratory. Each sample and sample preparation container shall be labeled with the sample identifier. If the sample identifier is different from the sample ID assigned at the field, it shall be cross-referenced to the sample ID.

5.3.2 Sample Handling

The following summarizes the general sample flow:

- Sample collection, packaging, and shipment,
- Sample receipt and analysis,
- Sample archiving, and
- Sample disposal

Table 13 identifies personnel primarily responsible for ensuring proper handling, custody, and storage or field samples during the above different stages of sample flow.

5.3.3 Sample Delivery

Unless specified in the SS-WP, samples will be delivered directly to the laboratory facility by overnight delivery service via common carriers (e.g., Federal Express and United Parcel Service Inc.). Samples will be grouped in sample delivery groups (SDGs) and each sample delivery group should contain 20 or fewer field samples within a project. An SDG signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated QA/QC samples. Samples may be shipped to the Laboratory in a single shipment or multiple shipments over a period of time, depending on the size of the SDG. A Sample Delivery Group (SDG) is defined by the following, whichever is most frequent:

- Each cooler of field samples received, or
- Each 20 field samples (including field QC samples) within an SDG, or
- Each 7 calendar day period (excluding Sundays and Government holidays) during which field samples in an SDG are received (said period beginning with the receipt of the first sample in the SDG).

Samples should be packaged, marked and labeled in accordance with the SAP (Section 16). Samples should be shipped in compliance with the most recent U.S. Department of Transportation regulations for shipping hazardous and nonhazardous materials, and in accordance with the analytical methodology. Shipment papers, including bills of lading and airbills, should be retained by the laboratory with chain-of-custody records. Chain-of-custody forms will be used as sample shipment forms.

5.3.4 Sample Custody

Sample custody procedures ensure accountability for the location and integrity of the sample at all times. Sample custody documentation for the project includes chain-of-custody (COC) forms, custody seals provided by the laboratory, laboratory sample receipt forms, laboratory sample transfer forms, traffic reports (e.g., air bills), and sample identification.

A COC record accompanies the sample container from the laboratory to the field where the sample is collected, preserved, and then returned to the laboratory. The field sampling team should neatly and clearly fill out the COC form provided by the laboratory. Special care should be used to differentiate the number zero from the letter “O”, the number five from the letter “S” and the number one from the letter “I”. Each cooler shipped to the laboratory should contain its own COC form. The field sampling team should file one copy of each COC in the project file, place the remaining copy (or copies) in a zip-top baggie and attach the baggie to the inside lid of the associated cooler. The laboratory’s sample custody program must meet the criteria listed below.

- The laboratory has designated a sample custodian who is responsible for maintaining sample custody and for maintaining all associated records documenting sample custody.
- Upon receipt of the samples, the custodian signs the COC record and records the date and time the samples are received. The custodian then measures and records sample temperature (using the temperature blank) on a cooler receipt form, checks for proper preservation, and checks the original COC documents and compares them with the labeled contents of each sample container for correctness and traceability. In the event any discrepancy is found, or the cooler temperature is outside the acceptable range of 2 to 4^{o}C, the laboratory should immediately contact Parsons PM as part of the corrective action process. Parsons PM will notify the Army if samples are received outside the above listed temperature range.

- A qualitative assessment of each sample container is performed to note any anomalies, such as broken or leaking containers. This assessment will be recorded as part of incoming COC procedures. In the event any sample containers are received compromised, the laboratory should immediately contact the Parsons PM as part of the corrective action process.
- The samples are stored in a secured refrigerator until analyses begin. Refrigerators will be maintained at 4 °C ± 2 °C, and the temperatures will be recorded daily.
- A copy of the COC and cooler receipt forms should be included in each laboratory data package.

Sampling packaging and shipment SOPs (including types of sample tags, labels, custody seals, and forms to be used, sample numbering system, and other sample handling and tracking information) are presented in Section 16.

5.3.5 Unused Sample and Extracts Storage

All samples should be submitted with more than enough volume for analysis (i.e., at least twice the volume required for analysis) and any remaining sample volume will be appropriately stored by the laboratory. The laboratory is required to retain unused sample volume and used sample containers for a period of 120 days after data submission. From the time of receipt until disposal, the laboratory shall maintain all samples and unused sample volumes at 4°C (±2°C) and protected from light. Samples and unused sample volumes must be stored separately from sample extracts and standards. The laboratory shall retain all sample extracts after analysis in bottles/vials with Teflon-lined septa and shall maintain stored extracts at 4°C (±2°C). The laboratory is required to retain the sample extracts for 180 days after data submission.

6 SCREENING ANALYTICAL METHODS

Screening or non-definitive analytical methods can be useful tools in generating quality environmental data. These methods should be selected as part of the overall systematic planning process and can serve to minimize sampling error, thereby minimizing costs. The various analytical screening methods that may be used for the project are shown in Table 3. Table 3 also presents a summary of reporting limits for screening methods. The methods and QC procedures were taken from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (USEPA SW-846, Third Edition, and its subsequent updates), *Methods for Chemical Analysis of Water and Waste* (USEPA 1979), *ASTM Annual Book of Standards* (1993), and from various manufacturers' literature. Specific methods for a project are to be selected from Table 3 unless a variance is requested. The list below is not intended to be all inclusive. Other appropriate methods based on site-specific PQOs will be provided in the site-specific work plan.

Methods acceptable to the NYSDEC will be utilized for the determination of the presence of free product in soil or water. Such method include, without limitation, visual identification of sheens or other visible product, measurable thickness of product on the water table, the use of field instruments, ultraviolet fluorescence, soil-water agitation, centrifuging, and hydrophobic dye testing (NYSDEC, 2002).

Field screening analysis should be conducted consistent with the NYSDEC DER-10, Section 2.1(g). In brief, field screening methods for all sampling matrices (soil, water, air, interior surfaces) can only be used for contaminant delineation if contaminant identity is known or if there is reasonable certainty that a specific contaminant may be present; or to bias sample location to the location of greatest suspected contamination. Field screening methods should not be used to verify contaminant identity or clean zones unless there has been an correlation study approved in advance by the Division of Environmental Remediation (DER) for the specific site where screening methods are proposed for verification. Where field screening is used, a standard operating procedure will be developed and a duplicate analysis of 10% of the samples will be conducted. Laboratory confirmation on 10% of the samples by a standard ASP method is required. There should be no bias in the selection of duplicate or laboratory confirmation samples, such as selecting positive detections for duplication or confirmation. The duplicate or confirmation analysis should be done on every 10^{th} sample, selected in the order they are presented for analysis. Laboratory confirmation occurs if the correlation between field screening and laboratory results are within +/- 30%. Analysis must be done by a Field Analyst with the following minimum qualifications: (1) Completion of a certification course or training by an experienced analyst who has demonstrated proficiency in the method; or, (2) Demonstration of the analyst's proficiency by correlation of the analyst's results with laboratory confirmation analysis.

7 DEFINITIVE DATA ANALYTICAL METHODS

This section presents sample preparation methods and analytical methods. The identified methods basically follow requirements and guidelines set out in the USEPA SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA Region 2 SOPs, and NYSDEC Analytical Services Protocol (2000). These methods have been developed specifically for the highly variable environmental samples and are reviewed and updated on a frequent basis in order to obtain the best possible quality data. Although specific method updates are noted in this document, the most recent updates to USEPA SW-846 and the NYSDEC ASP should be used if specified in the SS-WP.

As with the screening procedures in Section 6, the following methods and associated quality control requirements are subject to project-specific objectives developed during the DQO or systematic planning process. Once adopted, modification of these method-specific quality control and corrective action requirements involves appropriate communication and demonstration that the variances are adherence to PQOs and are consistent with the USEPA and NYSDEC ASP program goals. The ultimate goal is the generation of the highest quality defensible data necessary for informed decisions affording the decision-makers or stakeholders, a known and documented level of acceptable risk associated with the respective decision(s).

Section 7.1 presents brief description of preparation methods and Section 7.2 contains brief description of analytical methods. Table 6-A through Table 6-G present target analyte lists for various analytical methods and Table 7-A through Table 7-F present quality control requirements for various analytical methods. Table 8 summarizes performance criteria for surrogate recovery, Table 9-A through Table 9-F provides QC limits for LCS, Table 10-A through 10-C specifies QC criteria for MS/MSD results, and Table 12 presents performance criteria for field duplicates and laboratory duplicates.

7.1 SAMPLE PREPARATION METHODS

In accordance with the NYSDEC DER-10 guidance, sample matrix cleanup methods will be performed if:

1. Petroleum contaminated soils, sediments, or other solids are analyzed for semivolatile organics, and the method detection limits are elevated above the applicable remediation standard because of matrix interference;
2. Gas chromatographic peaks are not adequately separated due to matrix interference. A peak will be considered inadequately separated when a rise in baseline or extraneous peaks interfere with:
    - the instrumental ability to correctly identify compounds present (including internal standards and surrogates), and/or;
    - the integration of peak area and subsequent quantitation;
3. So specified by the analytical method; or
4. Matrix interferences prevent accurate quantification and/or identification of target compounds.

7.2 ANALYTICAL METHODS

Analytical methods should be identified based on site-specific information and objective and should be specified in the SS-WP. This SAP has identified several commonly used analytical methods for the project and the corresponding performance criteria are presented in Table 7-A through 7-F. Any variation from the methods identified in this SAP or selection of an appropriate method not recorded in the SW-846 or NYSDEC ASP should be documented in the site-specific work plan and a standard operating procedure should be developed and recorded. For tissue analysis, methods for each analyte to be tested will be proposed and approved by the NYSDEC.

For all petroleum storage and discharge areas, sample analysis should be conducted pursuant to the requirements of Spill Technology and Remediation Series (STARS) Memo #1 - Petroleum-Contaminated Soil Guidance Policy. Samples taken in non-petroleum storage and discharge areas should be analyzed for the stored material. Analysis should be conducted using any gas chromatography method by a laboratory that is certified pursuant to NYSDOH ELAP for the category of parameters being analyzed for.

7.3 ANALYTICAL SOPS

Table 4 presents analytical SOPs that will be used for the project. A NYSDEC ASP program, which contains SOPs for the referenced analytical methods, is attached in Appendix F.

7.4 TARGET COMPOUND

Unless specified in the site-specific work plan, for each analytical method, target compounds should include those listed in the Target Compound List (TCL) presented in NYSDEC ASP Appendix C. Target Compound List for various analytical methods are presented in Table 6-A through Table 6-G.

Tentatively identified compounds will be reported in the laboratory deliverables. If tentatively identified compounds or unknown compounds are detected at concentrations in excess of the applicable standards, criteria, and guidance (SCG), they should be addressed in either of two ways listed below. If a contaminant specific SCG does not exist for tentatively identified compounds and for unknown compounds, the generic SCG (class of contaminant, e.g. semi volatile compounds) should be used.

1. If the area will be remediated and it is likely that concentration of the tentatively identified compounds/unknown compounds will be reduced by the remediation, the tentatively identified compounds/unknown compounds should be analyzed in post remediation samples to document that they no longer exceed the applicable SCG.
2. An attempt should be made to positively identify and accurately quantify the tentatively identified compounds/unknown compounds using an analytical method consistent with this section so that a remediation standard can be developed.

7.5 TISSUE SAMPLING AND ANALYSIS

If tissue analysis is required, the following Quality Assurance procedures should be followed.

1. Analysis of lipid content is required for all organochlorine compounds.
2. For gas chromatography, detector systems other than mass spectrometers are required for identification and quantification of some analyte groups depending on the extraction method used during preparation of the tissue for analysis. Proposed methods should be proposed and approved prior to analysis.
3. General EPA quality control recommendations for tissue are contained in the NYSDEC DER-10, Appendix 2C. Alternate quality control requirements may be specified depending on the specific analysis being done.
4. The Quality Assurance Project Plan for tissue analysis should follow the outline in the USEPA publication "Preparation Aids for the Development of Category I Quality Assurance Project Plans" (EPA/600/8-91/003).
5. Tissue sampling should follow the current procedures for biota collection, preparation, and analysis as directed by the DER.

7.6 TOXICITY TESTING

If toxicity testing is required, the quality assurance procedures contained in the latest approved USEPA or ASTM methods or any method approved by the DER should be followed.

7.7 AIR SAMPLING

If air sampling is required, the SOPs specified in the method approved by the USEPA or/and NYSDEC for the sampling should be followed. Quality assurance procedures should follow the guidelines or direction of the USEPA and NYSDOH and should be recorded in the site-specific work plan.

8 DATA MANAGEMENT AND EVALUATION

The data reduction, verification, validation, assessment, and reporting procedures described in this section will ensure that: (1) the data are reviewed and documented; (2) transcription and data reduction errors are minimized; (3) complete documentation is maintained; and (4) the reported results are accurate, or qualified if necessary. Laboratory data reduction and verification procedures are required to ensure that the data deliverable(s) meet the overall project objectives. Data reduction, whether performed by instrumentation or manually, shall follow methodologies specified in the laboratory SOPs or approved analytical methods. Project-specific variations of general procedures, statistical approach, or formulas must be identified and be detailed in the SS-WP. Any variances from established procedures must be requested and approved in advance. Automated procedures shall be verified as required by USEPA's guidance on Good Automated Laboratory Practices (GALP, EPA 2185); all software shall be tested with a sample set of data to verify its correct operation via accurate capture, processing, manipulation, transfer, recording, and reporting of data. Data are reported in hardcopy data package(s) and as electronic data deliverable(s) (EDDs).

8.1 DATA REVIEW REQUIREMENTS FOR SCREENING DATA

Parsons will complete a 100 percent review of all screening data. The screening data methods are identified in Table 3. Calibration and QC requirements not within acceptable limits will be recorded. The calibration, QC requirements, and corrective action requirements required are shown in Table 3.

Screening data deliverables shall be prepared for all field analyses. The screening data performed at field shall be reported on the AFCEE screening data report forms (as attached in Appendix B). All field and QC sample results, calibrations, and calibration verifications should be recorded in a field logbook or the data report forms to ensure proper verification of sample results. Parsons QA officer will be responsible for the review of the entire screening data report package, including the associated field records. The results of this review shall (1) determine if the project objectives have been met, and (2) calculate the completeness of the screening data for the project. These results shall be included in the screening data deliverable.

8.2 DATA REVIEW LABORATORY REQUIREMENTS FOR DEFINITIVE DATA

Scientifically sound data of known and documented quality that meet project quality objectives are essential for use in the decision-making process. Data review is the process whereby data are examined and evaluated to varying levels of detail and specificity by a variety of personnel who have different responsibilities within the data management process. This section presents requirements for the laboratory to conduct review of definitive data.

8.2.1 Laboratory Data Reporting Requirements

The laboratory deliverables should be consistent with the NYSDEC ASP requirements, presented in Appendix B of the ASP. All deliverables will be in the CLP or CLP equivalent Format. The chemistry data package must contain adequate information and be presented in a clear, legible, concise, and consecutively paginated manner. The data package will include a sample data summary package and a sample data package. Data packages should be delivered in accordance with the schedule communicated from the project manager. Raw data (including electronic media) of all field

samples, QC samples, standards, and blanks should be archived and be available upon request for 5 years from the date of generation in accordance with the USEPA (2004) requirement.

8.2.1.1 Sample Data Summary Package

A Sample Data Summary Package shall be delivered separately (i.e., separated by rubber bands, clips or other means) directly preceding the Sample Data Package. Sample data forms shall be arranged in increasing project sample number order, considering both letters and numbers. The Sample Data Summary Package consists of copies of specified items from the Sample Data Package. The Sample Data Summary Package shall contain all data for all samples within one Sample Delivery Group of the Case and shall be ordered as follows.

1. NYSDEC Data Package Summary Forms
2. SDG Narrative
3. By fraction (VOA, SV, PEST, INORG, CONV) and by sample within each fraction - tabulated target compound results (Form I-ORG or Form I-IN) and tentatively identified compounds (Form I-ORG, TIC) (VOA and BNA only)
4. By fraction (VOA, SV, and PEST) - surrogate spike analysis results (Form II-ORG) by matrix (water and/or soil) and for soil, by concentration (low or medium)
5. By fraction (VOA, SV, and PEST) - matrix spike/matrix spike duplicate/matrix spike blank results (Form III-ORG) - as required by method.
6. By fraction (VOA, SV, and PEST) - QC Check Sample/Standard Recovery Summary - If required by method.
7. By fraction (INORG and CONV only) - duplicate sample results (Form VIIN)
8. By fraction (INORG and CONV only) - spike sample results (Form V-IN)
9. By fraction (VOA, SV, PEST, INORG, CONV) - blank data (Form IV-ORG and Form III-IN) and tabulated results (Form I-ORG and Form I-IN) including tentatively identified compounds (Form I-ORG, TIC)(VOA and BNA only).
10. By fraction (VOA and SV only) - internal standard area data (Form VIIIORG).

8.2.1.2 Sample Data Package

The Sample Data Package is divided into the following eight major units, if applicable: SDG case narrative, Contract Lab Sample Information Sheets, chain-of-custody forms, CLP volatiles data, CLP semivolatiles data, CLP pesticide/aroclor data, inorganic data. The CLP data for volatiles/semivolatiles/pesticide/aroclor data include QC summary, sample data, standards data, raw QC data, copy of calculations, and copy of extraction The inorganic data portion includes inorganic sample results, quality control data, verification of instrument parameters, raw data, copy of calculations, and digestion logs. The data package should be prepared consistent with the NYSDEC ASP and the forms specified in the NYSDEC ASP will be used for the data package. If the analysis of a fraction is not required, then that fraction-specific unit is not required as a deliverable.

The Sample Data Package shall include data for analyses of all samples in one Sample Delivery Group, including field samples, reanalyses, blanks, duplicates, spikes, matrix spikes, matrix spike duplicates, and matrix spike blanks.

The Laboratory shall retain a copy of the Sample Data Package for 365 days after final acceptance of data. After this time, the Laboratory may dispose of the package.

8.2.1.3 Case Narrative Requirements

An important part of the laboratory documentation is the case narrative. The case narrative contains essential information that affords an informed evaluation of data usability. The case narrative shall be clearly labeled "SDG Narrative" and shall contain: laboratory name and location, case number; Sample Delivery Group number; sample numbers in the SDG, differentiating between initial analyses and re-analyses; contract number; project name and site location; and detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing the samples reported in the data package.

Whenever data from sample re-analysis are submitted, the laboratory shall state in the SDG Narrative for each re-analysis, whether it considers the re-analysis to be billable, and if so, why.

The laboratory must also include any problems encountered: both technical and administrative, corrective actions taken, and resolution and an explanation for all data qualifiers (i.e. flags) applied to the data.

The SDG Narrative shall contain the following statement, verbatim: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on floppy diskette has been authorized by the Laboratory Manager or his designee, as verified by the following signature." This statement shall be directly followed by signature of the Laboratory Manager or his designee with a typed line below it containing the signer's name and title, and the date of signature.

The SDG Narrative itself must be signed in original signature by the Laboratory Manager or his designee and dated.

In summary, the following elements should be included in the case narrative:

- Cooler temperature, as required by the NYSDEC ASP.
- Table summarizing samples received, correlating field sample numbers, laboratory sample numbers, and laboratory tests completed.
- Discussion of sample appearance and integrity issues that may affect data usability (temperature, preservation, pH, sample container type or volume, air bubbles, multiphasic samples, excess headspace in soil VOC containers, the presence of multiple phases, etc.)
- Samples received but not analyzed and why.
- Discussion of holding time excursions for sample preparation and analyses.

- Analysis of all out-of-control or discrepancies of calibrations, continuing calibrations or QC sample results (surrogates, LCS, MS/MSD, post-digestion spikes, etc.), raw data/chromatograms and corrective actions taken.
- Identification of samples and analytes for which manual integration was necessary.
- Discussion of all qualified data and definition of qualifying flags.
- Discussion and recommendations of potential data usability of qualified data including detailed discussion of conditions associated with Q-flagged data.

8.2.1.4 Requirements for Reconstructed Total Ion Chromatograms

Reconstructed Ion Chromatograms (RIC) should be reported for each sample or sample extract. RICs must be normalized to the largest non-solvent component and contain the following header information:

- Sample number
- Date and time of analysis
- GC/MS instrument ID

Internal standard and system monitoring compounds are to be labeled with the names of compounds, either directly out from the peak, or on a printout of retention times if retention times are printed over the peak. If automated system procedures are used for preliminary identification and/or quantification of the target compounds, the complete data system report must be included in all sample data packages in addition to the reconstructed ion chromatogram. The complete data system report shall include all of the information listed below. For laboratories that do not use the automated data system procedures, a laboratory "raw data sheet", which contains the following information, must be included in the sample data package in addition to the chromatogram.

- Sample number
- Date and time of analysis
- RT or scan number of identified compounds
- Ion used for quantitation with measured area
- Copy of area table from data system
- GC/MS instrument ID
- Lab file ID

In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS operator must identify such edits or manual procedures by initialing and dating the changes made to the report.

8.2.1.5 Requirements for Reporting Compound Identification

For each sample, by each compound identified, the following shall be included in the data package:
a) copies of raw spectra and copies of background-subtracted mass spectra of target compounds listed in NYSDEC ASP that are identified in the sample and corresponding background-subtracted TCL

standard mass spectra. Spectra must be labeled with NYSDEC sample number, lab file ID, date and time of analysis, and GC/MS instrument ID; compound names must be clearly marked on all spectra. b) copies of mass spectra of organic compounds not listed in the Target Compound List (Tentatively Identified Compounds) with associated best-match spectra (three best matches).

8.2.1.6 Requirements for Reporting Compound Quantitation

The laboratory must provide a copy of the calculations work sheet showing how final results are obtained from values printed on the quantitation report. If manipulations are performed by a software package, a copy of the formula used must be supplied as well as values for all terms in the formula.

8.2.1.7 Reporting Limits Requirements

Reporting requirements associated with reporting limits are presented as follows:

- MDLs and sample results should be reported to one decimal place more than the corresponding RL, unless the appropriate number of significant figures for the measurement dictates otherwise.
- Soil samples shall have results reported on a dry weight basis. A wet weight aliquot of sample equivalent to the method specified dry weight aliquot of sample should be taken for analysis. Alternatively, the lab may choose to use a consistent wet weight aliquot that is expected to be large enough to compensate for the moisture in the sample (e.g., 50% more) and use this as a consistent weight.
- If possible, samples should be analyzed undiluted and non-detects reported to the project specified RLs. RLs for minority constituents in highly contaminated samples may have to be adjusted for dilutions.
- If the non-detect "ND", "U", "<", or other lower limit reporting convention is used, then these terms must be defined (EM200-1-6).
- RLs should be below the CRQL/CRDL, if applicable.

8.2.2 Manual Integrations

Manual integrations are an integral part of the chromatographic analysis process; they should be used judiciously to correct any incorrect integration by the automated instrumentation and not as a routine procedure for the purpose of meeting calibration or method QC acceptance criteria. Improper use of manual integrations (for example, peak shaving or peak enhancement) are considered improper, unethical, or illegal actions if performed solely to meet QC requirements. Manual integrations shall be done only as a corrective action measures. Examples of instances where manual integration would be warranted include, but are not limited to, co-eluting compounds resulting in poor peak resolution, a misidentified peak, an incorrect retention time, or a problematic baseline. When manual integrations are used, the following procedures are to be implemented for documenting the event and for consistency in performing the manual integration:

• The laboratory should provide SOP for manual integrations, if warranted. This SOP shall specify when automated integrations by the instrument are likely to be unreliable, what constitutes an unacceptable automated integration, and how the problems should be resolved by the analyst. This includes procedures for the analyst to follow in documenting any required manual integrations.

• When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations. The raw data records shall include the results of both the automated and manual integrations (i.e., "before" and "after" chromatograms of manually integrated peaks), notation of the cause and justification for performing the manual integrations, and date, and signature/initials of person performing the manual operations.
• All manual integrations must be reviewed and approved by the laboratory Section supervisor and/or the laboratory QA officer.
Note: Both the primary and secondary reviews (analyst's and supervisory) may be performed electronically, provided all documentation and data integrity are maintained.
• All manual integrations must be identified in the case narrative. This will ensure consistency when manual integrations are performed and facilitate review and acceptance of manually integrated data.

8.2.3 Tentatively Identified Compounds

Tentatively identified compounds (TICs) are compounds not associated with the calibration standards which are identified in methods with MS detection. All peaks with a response greater than 10% of the nearest internal standard are potential TICs and should be examined and reported. Qualitative identification of TICs is performed by computer searches of standard reference libraries and TICs may be reported as a specific chemical or as a member of a chemical family. For each volatile sample, the Laboratory shall conduct a search to determine the possible identity of up to 10 organic compounds of greatest concentration which are not system monitoring compounds or internal standards and are not listed as volatile TCL. For each semivolatile sample, the Laboratory shall conduct a search to determine the possible identification of up to 20 organic compounds of greatest concentration, which are not surrogates or internal standards and are not listed as semivolatile TCL. In performing searches, the NIST/EPA/NIH (May 1992 release or later) and/or Wiley (1991 release or later), or equivalent, mass spectral library shall be used. Concentrations are estimated assuming a response factor of 1 between the TIC and the nearest internal standard.

8.2.4 Laboratory Data Review Requirements

All analytical data generated by the laboratory shall be verified prior to submittal to Parsons. This internal data review process, which is multi-tiered, shall include all aspects of data generation, reduction, and quality control assessment. Procedures for laboratory verification and validation of data should be summarized in the laboratory QA manual. Each result reported by the laboratory should undergo multiple levels of internal data review. The analysts and technicians provide primary data review for 100 percent of the definitive data at the bench level, secondary review should be performed by independent experienced quality control personnel on 100 percent of the data, and the final data packages are reviewed by the laboratory's section supervisor, QA manager, customer service representative or project contact before submission to Parsons.

The following elements for review/verification at each level must be included, but not be restricted to, in the review conducted by the laboratory:

• Sample receipt procedures and conditions,
• Sample preparation,
• Appropriate SOPs and analytical methodologies,
• Accuracy and completeness of analytical results,
• Correct interpretation of all raw data, including all manual integrations,

• Appropriate application of QC samples and compliance with established control limits.
• Verification of data transfers,
• Documentation completeness (e.g., all anomalies in the preparation and analysis have been identified, appropriate corrective actions taken, and have been documented in the case narrative(s), associated data have been appropriately qualified, anomaly forms are complete), and
• Accuracy and completeness of data deliverables (hard copy and electronic).

8.2.5 Laboratory Data Evaluation

The calibration, QC, and corrective actions for definitive data are shown in Tables 7-A through Table 12. Data qualifiers shall be applied by the laboratory as part of their validation activities. The data qualifiers for definitive data should be specified in the data deliverable package. Flagging criteria apply when acceptance criteria are not met and corrective actions were not successful or not performed. The data qualifiers are reviewed by the supervisor of the respective analytical sections after the first and second level reviews of the laboratory data have been performed. No qualifiers will be applied to TICs.

The laboratory QA section shall perform a 100 percent review of 10 percent of the completed data packages, and the laboratory project representative shall complete a final review on all the completed data packages.

Parsons will subsequently evaluate the flags applied by the laboratory as part of the data validation and usability assessment activities. The flags may be accepted, modified, or rejected. For all data qualifiers that are changed, Parsons will provide clear justification for those modifications based on project-specific quality objectives.

8.2.6 Method Blank Evaluation Guidance

The following criteria shall be used to evaluate the acceptability of the blank data, unless project quality objectives specify otherwise. For method blanks, the source of contamination shall be investigated and measures shall be taken to correct, minimize, or eliminate the problem if the concentration exceeds the RL (Use the limits specified in Table 7-A through 7-F for common laboratory contaminants.). If the RL is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary, based on the following criteria: i) the method blank contamination relative to the measured concentration of any sample in the associated preparation batch, or ii) there is evidence the blank contamination otherwise affects the sample results. Except when the sample analysis resulted in a non-detect, all samples associated with method blank contamination and meeting these criteria shall be reprocessed in a subsequent preparation batch. If no sample volume remains for reprocessing, the results shall be reported with appropriate flag, along with any other appropriate data qualifier. If an analyte is found only in the method blank, but not in any batch samples, no flagging is necessary. Method blanks should also be examined to verify that any TICs present in the samples are not found in the blank. Method blank contamination must be addressed in the case narrative.

8.2.7 Laboratory Data Reduction

Data reduction is the process by which raw analytical data generated from laboratory instrument systems are converted into usable concentrations. The raw data, which may take the form of area

counts, instrument responses or observations, are processed by the laboratory and converted into concentrations expressed in the parts-per-million (ppm) or parts-per-billion (ppb) range. Raw data from these systems include compound identifications, concentrations, retention times, and data system printouts. Raw data are usually reported in graphic form, bargraph form, or tabular form. The laboratories will follow the applicable data reduction SOPs for data reduction requirements. Concentration units are to be listed on reports and any special conditions, such as dry weight conversions will be noted. "Non-detects" will be reported as less than the PQL. Results reported greater than the MDL but less than the PQL will be reported as estimated and flagged by the laboratory.

8.3 DATA TRANSORMATION AND DATA REDUCTION

Field personnel will record all field data in bound field notebooks and on standard forms. During processing of field data, validation checks will be performed by individuals designated by the project manager. The purpose of these checks is to identify "outliers;" that is, data which do not conform to the pattern established by other observations. Because of the limited number of observations, detailed statistical analysis of the data to be obtained during this project is not feasible, and the principal method of validation will be routine checks to assure that data are correctly transcribed and that reported identification codes and sampling information match the corresponding information in the field records. In addition, data will be compared against those obtained in previous investigations (where available) and against applicable standards and guidelines.

Although outliers may be the result of transcription errors or instrumental breakdowns, they may also be manifestations of a greater degree of spatial or temporal variability than expected. Therefore, after an outlier has been identified, a decision must be made concerning its further use. Obvious mistakes in data will be corrected when possible, and the correct value will be inserted. If the correct value cannot be obtained, the data may be excluded. An attempt will be made to explain the existence of the outlier. If no plausible explanation can be found for the outlier, it may be excluded, but a note to that effect will be included in the report. Also, an attempt will be made to determine the effect of the outlier when both included and excluded in the data set, and the results will be discussed in the report.

After checking the validity of the data in the field notes, the field team leader or his designee will reduce the data to tabular form, when possible, by entering the data into data files. Where appropriate, the data files will be set up for direct input into the project database. At a minimum, 10% of the data entered into the database will be verified through a QC process. Subjective data will be filed as hard copies for later review by the project manager and incorporation into technical reports, as appropriate.

Sample calculations are contained in the method specifications. All concentration data shall be expressed in units of micrograms per liter (ug/L) or micrograms per kilogram (ug/Kg) dry weight, as appropriate for the matrix. The field measurements of pH, conductivity, turbidity, and temperature shall be reported in standard logarithmic, umho/cm, nephelometric turbidity units (NTUs), and degrees Celsius, respectively. All definitive analytical values and screening measurement values should be reported to appropriate significant figures consistent with the measurement. As an example, all water levels measured in wells will be reported to the nearest 0.01 foot and soil sampling depths will be reported to the nearest 0.1 foot.

All analytical results are carefully reviewed and formatted into final submittal form by experienced quality control personnel. The data will be input into the project database, as described in Section 8.12.2.

8.4 DATA ASSESSMENT PROCEDURES

Scientifically sound data of known and documented quality that meet project quality objectives are essential for use in the decision-making process. Data assessment is the process whereby data are examined and evaluated to varying levels of detail and specificity by a variety of personnel who have different responsibilities within the data management process. For definitive data, the data assessment includes data verification, validation, and usability assessment. For screening data, data verification will be conducted to ensure data quality. There must be persuasive records that document data review activities to afford effective assessment of the data for its quality and usability. The data can then move forward with associated qualifiers indicating the overall usability of the data.

Data verification is the first step in data review. As used here, data verification is confirmation that the specified requirements have been performed, i.e., it is a completeness check. The detailed discussion of data verification is presented in Section 8.5.

Data validation extends this and is confirmation that the requirements for a specific intended use are fulfilled. Data validation is the systematic process of evaluating the compliance of the data with the pre-defined requirements of the project, including method, procedural, or contractual requirements and the comparison of the data with criteria based on the quality objectives documented in this SAP and the SS-WP. The purpose of data validation is to assess the performance associated with the analysis in order to determine the quality of the data. Data validation includes a determination, to the extent possible, of the reasons for any failure to meet performance requirements, and an evaluation of the impact of such failures on the usability of the data. Data validation procedure is presented in Section 8.6.

The data usability assessment is an evaluation based on the results of data validation and verification in the context of the overall project decisions or objectives. The assessment determines whether the project execution and resulting data meet project quality objectives. Both the sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data. The requirements for data usability assessment are presented in Section 8.7.

8.5 DATA VERIFICATION

Data verification is the most basic assessment of data. Data verification is a process for evaluating the completeness, correctness, consistency, and compliance of a data package against a standard or contract. In this context, "completeness" means all required hardcopy and electronic deliverables are present. Data verification will be performed by Parsons for all laboratory delivered data and field screening data.

8.6 ANALYTICAL DATA VALIDATION

Based on the information in the data package, a reviewer should be able to determine the precision, accuracy, representativeness, completeness, comparability, sensitivity, and defensibility of the data.

Data validation for laboratory data will be performed for all definitive sample results in accordance with the requirements contained in the analytical method, the SAP and site-specific work plan, the NYSDCE ASP, the USEPA Region 2 SOPs, the USEPA National Functional Guidelines for Data Review (USEPA, 1999, 2004). Data validation will be manually performed by the project chemist or personnel trained by the project chemist. The project chemist will review at least 20% of the data validated by the trained personnel and is responsible to oversee the whole data validation process. In performing the data validation, the raw data are spot-checked in accordance with the Region 2 SOP to evaluate whether there is any transcription error. The review of laboratory data will focus on the following subjects, if applicable:

- COC forms,
- Holding times, sample preservation, and sample conditions (e.g., percentage of solids),
- Instrument calibration and performance,
- Method blanks, trip blanks, equipment/rinsate blanks,
- Method detection limits and laboratory-established reporting limits,
- Analytical batch control records including laboratory spike recoveries and spike duplicate results, and matrix spike recoveries and spike duplicate results,
- Surrogate standard recoveries,
- Internal standard areas and RTs,
- Confirmation results for explosives,
- Chromatograms and mass spectrums,
- Corrective actions,
- Formulas used for analyte quantitation,
- Laboratory and field duplicate results,
- Calculations supporting analyte quantitation,
- ICP serial dilution,
- interference check sample results,
- ICP linear range, and
- Completeness of data.

Data outliers that fall outside of the QC criteria outlined in this SAP or site-specific work plan (e.g., Tables 5-A/B, Table 7-A through Table 7-F, Table 8, and Table 9-A through Table 9-F, Table 10-A through 10-C, and Table 12) will be flagged with an appropriate qualifier consistent with the USEPA Region 2 SOPs. All data validation flags applied will be added to the validated data with explanation prior to submittal. Data validation flags are provided in Table 17-A and Table 17-B for inorganics and organics, respectively. An example of the form that will be used for the data validation is provided in Table 18.

8.7 DATA USABILITY ASSESSMENT

A usability assessment evaluates whether data meet project quality objectives as they relate to the decision to be made, and evaluates whether data are suitable for making that decision. All types of

definitive data (e.g., sampling, on-site analytical, off-site laboratory) are relevant to the usability assessment. The usability assessment is the final step of data review and can be performed only on data of known and documented quality (i.e., verified and validated data).

A data usability assessment report will be submitted to the project manager by the Parsons project chemist to summarize the usability of the validated data. The report will include:

- A summary of data validation results,
- Overall data usability and completeness,
- Evaluation of each data quality indicator (whether meet the criteria, what potential impacts on data usability),
- Any deviations (e.g., holding time, QC performance criteria, sample location, sample collection SOPs) from the SAP and/or the site-specific work plan and the impact of deviations on the usability of data,
- Any problems with documentation or custody procedures and the impact on the usability of data,
- Damaged samples and the usability of the associated data, and
- Any other relevant issues.

8.8 NON-DIRECT MEASUREMENT DATA EVALUATION

Site-specific non-direct measurement data evaluation will be specified in the site-specific work plan. Non-direct measurement data that will be collected for the project include

- Site data from all previous investigations, and
- measurements that are ancillary to addressing the project's objectives (e.g., meteorological data)

Existing data will be evaluated in combination with newly collected data. An evaluation consistent with the USEPA QA/G-5 (2002) and USEPA (2004) Uniform Federal Policy for Quality Assurance Project Plans will be conducted to assess whether existing data meet the current project's acceptance criteria before the existing data are used for decision-making and will be recorded in project-specific work plan.

8.9 RECONCILIATION WITH USER REQUIREMENTS

Project results will be reconciled with the requirements defined by the data user or decision maker. Based on site-specific DQOs, the approach of data reconciliation will be discussed in site-specific work plan or SAP. Limitations on the use of the data will be reported in the project technical report.

8.10 ELECTRONIC DATA REPORTS

The laboratory shall provide an electronic data deliverable in the Environmental Restoration Program Information Management System format as specified in the AFCEE Guidance for Contract Deliverables, Appendix C - Quality Assurance Project Plan (QAPP, Version 4.0, 2005).

ERPIMS is a data management system designed to accommodate all types of data collected for AFCEE Installation Restoration Program. Specific codes and data forms have been developed to

allow consistent and efficient input of information to the system. The database information shall be provided by the laboratory via ASCII files in specified ERPIMS format. Electronic data reporting formats and requirements are given in the most current version of the *ERPIMS Data Loading Handbook*.

The laboratories will also submit a hard copy of the analytical data for environmental, field and laboratory QC samples. The electronic data delivery shall contain the same information as described for the hard copy deliverable. Electronic deliverables should be reported with no discrepancies from the hard copy. In general, the EDD submittal will include:

- the laboratory's identification of each field sample,
- field sample identifications,
- analytes,
- results,
- data qualifiers and validation flags,
- concentration units, and
- applicable QC data.

Additionally, the calibration information should be included in the EDD if the laboratory has that capability.

The project technical data other than the chemical analysis results such as site information; well characteristics; hydrogeologic, geologic, and physical analysis results will be recorded by Parsons as electronic files under the project directory.

8.11 PROJECT DATA TRACKING AND ARCHIVING

This section presents information on project data tracking (Section 8.11.1), archiving (Section 8.11.2), and storage and retrieval (Section 8.11.3).

8.11.1 Data Tracking

Project manager will be responsible for tracking data as they are collected, transformed or reduced, transmitted, and analyzed. Reports produced during each of the above phase will be submitted to project manager and archived in project files to ensure the data are properly tracked, reviewed, and validated for use.

8.11.2 Data Archiving

This section presents archiving procedures for electronic data (Section 8.11.2.1) and hardcopy data (Section 8.11.2.2).

8.11.2.1 Electronic Data Archiving

Electronic data shall be archived in project files and in electronic format by Parsons and the laboratory for the duration of the project or a minimum of five years, whichever is longer, or as dictated by project requirements (if longer than five years). The laboratory shall also provide for

Parsons and AFCEE all files associated with the project in electronic media. The data packages must be retrievable for AFCEE within seven calendar days. In the event of laboratory closure, all applicable documents and electronic media must be immediately transferred to AFCEE.

The laboratory shall maintain electronic records sufficient to recreate each analytical event conducted pursuant to the SOW. The minimum records the laboratory shall keep contain the following: (1) COC forms, (2) initial and continuing calibration records including standards preparation traceable to the original material and lot number, (3) instrument tuning records (as applicable), (3) method blank results, (4) IS results, (5) surrogate spiking records and results (as applicable), (6) spike and spike duplicate records and results, (7) laboratory records, (8) raw data, including instrument printouts, bench work sheets, and/or chromatograms with compound identification and quantitation reports, (9) corrective action reports, (10) other method and project required QC samples and results, and (11) laboratory specific written SOPs for each analytical method and QA/QC function in place at the time of analysis of project samples.

Parsons uses Windows (2000 or more recent version) system to perform electronic file operation and Oracle database or other appropriate programs to perform chemical analysis data management on network computers. The software programs are commonly used and upgraded whenever software changes occur. Parsons performs scheduled electronic data backups of project files and performs periodic archiving of electronic media on a scheduled basis. Electronic project files are maintained on a no-fault server; a no-fault server minimizes data loss during hard-drive failure by operating and distributing data sequentially over four separate physical hard drives. Back-ups of project files on to magnetic tapes on the no-fault server are performed on a weekly basis and updated daily, Monday through Thursday, through a differential back-up. A differential back-up replaces backed-up files that are edited between each daily update differential back-up.

Electronic tape back-ups are stored in a fire proof box either at Parsons or at an off-site storage location. Weekly backups onto magnetic tape are retained for a minimum of three weeks prior to overwriting; however, the last back-up each month is retained without being overwritten.

8.11.2.2 Hard Copy Data Archiving

Hardcopy data shall be archived in project files by Parsons and the laboratory for the duration of the project or a minimum of five years, whichever is longer, or as dictated by project requirements (if longer than five years). The laboratory shall maintain hardcopy records sufficient to recreate each analytical event conducted pursuant to the SOW.

All field measurements and instrument check data will be entered into an electronic database where it will also be maintained. In addition, hardcopy of field measurements and field notes will be archived in project files by Parsons for the duration of the project or a minimum of five years, whichever is longer, or as dictated by project requirements (if longer than five years).

8.11.3 Data Storage and Retrieval

All hardcopy and electronic chemical analysis data, field sheets, log books, and other relevant field documents (e.g., health and safety meeting sign-in sheets, personnel daily frisking forms, daily instrument check sheets) will be maintained by Parsons at Parsons or at an off-site storage location. If stored, the data packages will be retrievable within seven calendar days.

8.12 HARDCOPY DATA REPORT FORMS

The hardcopy data reports or forms shall conform to the formats identified in the NYSDEC ASP program. The NYSDEC ASP forms shall be used unless a variance is requested and approved in advance and that the forms included in the site specific work plan or SAP, can be verified that they contain at a minimum the information requested on the NYSDEC ASP forms. A complete list and description of forms is provided in the NYSDEC ASP. Other forms shall be included in the site-specific work plan, as needed.

For all analyses, at a minimum, the laboratory report will show traceability to the sample analyzed and will contain the elements presented below.

- Case narrative (identifies problems and corrective actions);
- Copy of signed COC;
- Cooler receipt forms documenting the date, time of receipt, condition of samples (including preservation) and labels, temperature of the shipping container, and verification of integrity of the custody seals;
- Laboratory name;
- Client name;
- Date of sample collection;
- Date of sample receipt;
- Date of sample extraction or preparation;
- Date of issue;
- Project name and unique identification number;
- Field sample name/number;
- Laboratory sample number;
- Sample matrix description;
- Analytical method description and reference citation for all analyses, preparation, cleanup procedures;
- Preparation, analysis and other batch numbers;
- Individual parameter;
- Analytical results with correct number of significant figures;
- All confirmation data, when performed;
- Date of analysis (first run and subsequent runs);
- Analysis time;
- Method reporting limits adjusted for sample-specific factors (i.e., aliquot size, dilution/concentration factors, moisture content;
- Method detection limits;
- Concentration units;
- Any data qualifiers assigned;
- Percent moisture or percent solids (all soils reported on dry weight basis);
- Any special conditions;
- Chromatograms, as needed;
- Sample aliquot analyzed;
- Final extract volume;

- Dilution or concentration factors (if dilutions result in non-detect values for all other analytes which showed detected concentrations in previous analyses, the results of both runs will be reported with the appropriate notations in the narrative);
- Initial and continuing calibration results;
- A cross-reference to identify applicable laboratory QC samples with field samples; and
- Corresponding QC summary report.

The laboratory reports should conform to the requirements presented in Section 8.2.1. QC data will be recorded on Contract Laboratory Program or CLP-equivalent QC summary forms for the appropriate tests and correlated to the analysis results by the laboratory lot control numbers. The QC results are used to prepare control charts for each test and matrix type. QC reports will contain the following items as appropriate:

- Narratives describing any non-compliant samples,
- Method blank, trip blank, equipment rinsate blank,
- Surrogate results,
- LCS/LCSD results,
- MS/MSD or MS/MD results, and
- Tuning results.

The laboratory will, as a part of the data reduction and validation process, confirm that its documentation is complete, sequentially paginated, and legible; qualitative identifications are accurate; calculations are accurate; and results are expressed in the appropriate units. The laboratory will also confirm that data documentation has been approved by the laboratory manager or designee.

Manual recording should be conducted legibly in ink, initialed and dated by the responsible person. Data should be corrected manually, if warranted, by using single line drawn through errors, initialed and dated by the responsible person.

8.13 DATA ANALYSIS

Parsons uses windows (2000 or more recent version) system to perform general file/data processing and the Oracle database to perform chemical analysis data management on network computers. In addition, various software and/or computer codes will be used at different project stages for different data analysis purposes. The following lists some of the commonly used software/computer codes for the project:

- XLSTAT (version 6.1.9 by Addinsoft), used for background comparison or any other statistical comparison;
- The computer code AQTESOLV™ (Geraghty & Miller, 1994) or similar, and the method of Cooper *et al*. (1967) for confined aquifers or the method of Bouwer and Rice (1976) and Bouwer (1989) for unconfined conditions, slug testing data analysis;
- The USEPA Software for Calculating Upper Confidence Limits (ProUCL version 3.00.02), risk assessment Exposure Point Concentration estimation;
- The Integrated Exposure Uptake Biokinetic Model for Lead in Children (IEUBK) developed by USEPA, risk assessment for child lead exposure;

- The Recommendations of the Technical Review Workgroup for Lead for an Interim Approach to Assessing Risks Associated with Adult Exposures to Lead in Soil (USEPA, 2003), risk assessment for adult industrial worker.

Software/computer codes used for project will be recorded in project technical document and PM and technical personnel are responsible for identifying the appropriate software for the project and for using the most recent version of the software.

Detailed discussion of system backup can be found in Section 8.11.

9 PERFORMANCE ASSESSMENT AND SYSTEM AUDITS

Audits will include a careful evaluation of both field and laboratory quality control procedures. Audits of field procedures will be performed before or shortly after systems are operational. The audits will be conducted by an individual who is technically knowledgeable about the operation(s) under review. This section discusses procedures for both performance audits (Section 9.1) and system audits (Section 9.2).

9.1 PERFORMANCE AUDIT PROCEDURES

Performance audits are conducted by introducing control samples into the data production process. These control samples may include performance evaluation samples, field samples spiked with known amounts of analyte, and split field samples that are analyzed by two or more analysts within or without the organization.

9.1.1 Laboratory Performance Audits

In addition to conducting internal reviews and audits, as part of its established Quality Assurance program, the laboratory is required to take part in regularly scheduled Performance Evaluations and laboratory audits from State and Federal agencies. These are conducted as part of certification processes and to monitor the laboratory performance. The laboratory shall use the information provided from these audits to monitor and assess the quality of its performance. Problems detected in these audits shall be reviewed by the laboratory Quality Assurance Manager and laboratory management and corrective action shall be instituted as necessary.

The laboratory will be required to conduct an analysis of Performance Evaluation (PE) samples or provide proof that Performance Evaluation samples submitted by USEPA or a state agency have been analyzed within the past twelve (12) months.

9.1.2 Field Performance Audits

Unless specified by the site-specific work plan, field performance audits will not be conducted for this project. Field performance will be assessed using QA/AC results (e.g., trip blank, equipment/rinsate blank, field replicate analyses, sample condition upon receipt by the laboratory). Each blank analysis will be considered an indirect audit of the effectiveness of measures taken in the field to ensure sample integrity (e.g., field decontamination procedures). The results of the field replicate analyses are an indirect audit of the ability of each field team to collect representative sample portions of each matrix type. In addition, Parsons QA Officer will be responsible to review in detail field procedures and field logs to verify compliance.

9.2 SYSTEM AUDIT PROCEDURES

Systems audits are qualitative inspections and reviews of the quality assurance system used by some part of or the entire measurement system. The audits are performed against a set of requirements, which may be a quality assurance project plan or work plan, a standard method, or a project statement of work. The primary objective of the systems audits is to ensure that the QA/QC procedures are being followed.

Field and laboratory quality control procedures and associated documentation may be system audited. These audits will be performed once during the performance of the project. However, if conditions adverse to quality are detected or if the project manager requests, additional audits may occur.

System audits will also be performed by data users including USEPA Region 2, AFCEE, NYSDEC, and the Army. Generally, the audit covers the SAP development and approval and SOP development and approval.

9.2.1 Laboratory Systems Audits

As part of its Quality Assurance Program, the Laboratory Quality Assurance Manager shall conduct periodic checks and audits of the analytical systems. The purpose of these is to ensure that the analytical systems are working properly and that personnel are adhering to established procedures and documenting the required information. These checks and audits will also assist in determining or detecting where problems are occurring.

The laboratory Quality Assurance Manager will periodically review laboratory control samples. These samples will reflect the quality of the entire analytical method, the efficiency of the preparation method and the analytical instrument performance. When a problem is detected, the Quality Assurance Manager will assist the analyst and laboratory management in determining the reason and in developing a solution. Rechecking of systems will be conducted by the Quality Assurance Manager as required.

Parsons QA officer or his/her designee is responsible for reviewing the laboratory QA/QC manual and ensure the laboratory QA/QC procedures are consistent with the project SAP requirement.

9.2.2 Field System Audit Procedures

System audits of field activities will be accomplished by an inspection of all field site activities. Field system audit should be conducted at the beginning of any long-term field sampling program (i.e., >1 week) and will be conducted on an ongoing basis during the project as field data are generated, reduced, and analyzed. Field audits, if warranted, should be specified in the SS-WP.

During the field audit, the auditor(s) will compare current field practices with standard procedures. The following elements will be evaluated during a field system audit:

- All activities including sample collection, equipment calibration, decontamination, record keeping conducted in accordance with the generic SAP and/or site-specific work plan;
- All procedures and analyses conducted according to procedures outlined in the generic SAP and/or site-specific work plan;
- Sample documentation;
- Working order of instruments and equipment;
- Level of QA conducted per each field team;

- Contingency plans in case of equipment failure or other event preventing the planned activity from proceeding;
- Decontamination procedures;
- Level of efficiency with which each team conducts planned activities at one site and proceeds to the next; and
- Sample packaging and shipment.

All numerical manipulations, including manual calculations, will be documented. All records of numerical analyses will be legible, of reproduction-quality, and sufficiently complete to permit logical reconstruction by a qualified individual other than the originator. After completion of the audit, any deficiencies will be discussed with the field staff and corrections implemented. If any of these deficiencies could affect the integrity of the samples being collected, the auditor(s) will inform the field staff immediately, so that corrections will be implemented immediately. The audit will be performed by the project QA officer, project chemist, field team leader, or designees. A standard form of field audit report and field daily quality control report is provided in Appendix B.

Field system audit may also be conducted by regulators.

9.3 AUDIT REPORT

Audit reports will be written by auditors who have performed the audit after gathering and evaluating all data. Items, activities, and documents determined by lead auditors to be in noncompliance shall be identified at exit interviews conducted with the involved management. Noncompliances will be logged and documented through audit findings, which are attached to and are a part of the integral audit report. These audit-finding forms are directed to Parsons project manager, the Army, and the regulators (contact information see Section 3) within fifteen days after the completion of the audit. Serious deficiencies will be reported to the project manager within 24 hours to satisfactorily resolve the noncompliance in a specified and timely manner. All audit checklists, audit reports, audit findings, and acceptable resolutions are approved by the QAO prior to issue. Corrective actions should be followed if any noncompliance is noted in the audit report. Verification of acceptable resolutions may be determined by re-audit or documented surveillance of the item or activity. Upon verification acceptance, the QAO will close out the audit report and findings.

10 PREVENTATIVE MAINTENANCE

A preventative maintenance program is necessary to help prevent delays in project schedules, poor output performance or erroneous results in investigative operations. Preventative maintenance on laboratory analytical equipment used in this project will be performed contractually by qualified personnel. Maintenance of field equipment will be performed routinely for sampling events. More extensive maintenance will be performed based on hours of use, by a qualified servicing organization. Repairs, adjustments and calibrations will be recorded.

10.1 FIELD EQUIPMENT

The three elements of the field equipment maintenance program include normal upkeep of equipment, service and repair (when required), and formalized record-keeping of all work performed on each piece of equipment. This section addresses the normal equipment upkeep element of the maintenance program. For most of the equipment, normal maintenance will consist of cleaning outside surfaces, lubrication of all moving parts, and, if applicable, a battery level check and recharge or replacement as necessary. This program will include the maintenance of all monitoring, measuring, and test equipment returning from use or any equipment used on a daily basis. The frequency of maintenance checks will be dependent on the individual needs and use of each piece of equipment. Maintenance procedures will be only those necessary for keeping an instrument in service or in preparation for everyday use. It is beyond the scope of this document to cover repair procedures for each piece of equipment. Repair problems will be referred to the manufacturer or other qualified servicing organization.

The field team leader, or the designated personnel, will be responsible for keeping all maintenance records, making sure all equipment used is maintained properly, informing field team members of any specific maintenance requirements for equipment used at the site and shipping any instrument in need of repair to the correct source.

The field personnel responsibilities include maintaining each piece of equipment located at the site and the maintenance of equipment after use. A record of equipment maintenance and repair will be kept in the field logbook.

Table 11 summarizes requirement for field equipment calibration, maintenance, testing, and inspection. These requirements are also briefed discussed in the following sections.

10.1.1 Field Equipment Calibration

The frequency of calibration for field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate, but daily as a minimum. To ensure comparability between sample data of similar samples and sample conditions, standard solutions and material traceable to the National Institute of Standards and Technology or EPA-published standards/protocols will be used to calibrate the field instruments. Table 11 summarizes requirement for field equipment calibration.

10.1.2 Field Equipment Inspection

Equipment to be used during field sampling will be examined to certify that it is in proper operating condition. This includes checking the manufacturer's operating manual and the instructions for each equipment to ensure that all maintenance requirements are being observed. Field notes for previous sampling trips will be reviewed so that the notations on any prior equipment problem are not overlooked and all necessary repairs to equipment have been carried out.

10.1.3 Field Equipment Maintenance

Equipment, instruments, tools, gauges, and other items requiring preventive maintenance will be serviced in accordance with the manufacturer's specified recommendations and written procedures developed by the operators.

Manufacturer's procedures identify the schedule for servicing critical items in order to minimize the downtime of the measurement system. It will be the responsibility of the field team leader to adhere to the maintenance schedule and to arrange any necessary and prompt service as required. Service to the equipment, instruments, tools, gauges, etc., will be performed by qualified personnel. In the absence of any manufacturer's recommended maintenance criteria, a maintenance procedure will be developed by the operator based upon experience and previous use of the equipment.

Logs will be established to record maintenance and service procedures and schedules. All maintenance records will be documented and traceable to the specific equipment, instruments, tools, and gauges.

Critical spare parts for field equipment will be located in the Parsons office at the Seneca Depot (Building 125). Records documenting field equipment calibration, maintenance, testing, and inspection activities will be archived under project file.

10.2 RENTAL EQUIPMENT

Rental equipment used on the project should be obtained only from a certified rental supplier. The equipment will require a prereceipt to verify accuracy, maintenance and upkeep of the equipment. A receipt indicating that the equipment has been checked upon return will be required as well.

10.3 LABORATORY INSTRUMENT

10.3.1 Laboratory Instrument Calibration

All laboratory instrument shall be calibrated in accordance with USEPA SW-846 analytical methodology and the requirements of the New York ASP.

10.3.2 Laboratory Instrument Maintenance

An important factor in maintaining accuracy and precision, achieving required holding times, and addressing contract schedule is preventive maintenance. As part of the laboratory's maintenance program, service contracts are held on critical analytical instruments. SOP's for routine maintenance of laboratory equipment are included as part of the laboratory QA manual and will be reviewed by project chemist before project starts. The SOPs submitted by the laboratory describe the procedures and documentation activities that will be performed to ensure that all analytical instrumentation and equipment are available and in working order when needed. The SOPs also discuss the ability to ensure that project schedules are met (e.g., availability of spare parts or spare instruments, instrument control (on-site and during storage), security, and availability (e.g., log-in/log-out procedures)).

Instrument and equipment maintenance logs must be kept to document analytical instrumentation and equipment maintenance, testing, and inspection activities.

11 NONCONFORMANCE/CORRECTIVE ACTIONS

A nonconformance is defined as an identified or suspected deficiency in an approved document, such as a technical report, calculation, or computer program; an item where the quality of the end item itself or subsequent activities using the document or item would be affected by the deficiency; or an activity that is not conducted in accordance with the established plans or procedures. When a significant condition adverse to quality is noted at the site or laboratories by the field staff and/or Project Chemist, the cause of the condition will be determined and corrective action taken to preclude possible repetition. Condition identification, cause, reference documents, and corrective action planned will be documented and reported to the Parsons PM, Parsons QA officer, the USACE Project Chemist, and involved subcontractor management. Implementation of corrective actions will be verified by documented follow-up action. All project personnel have the daily responsibility to promptly identify and report any condition adverse to quality, as well as to solicit the approved corrective action.

Parsons project manager has overall responsibility to ensure that all corrective actions necessary to resolve audit findings are acted upon promptly and satisfactorily. The project manager shall ensure that no further work dependent on the nonconforming item or activity is performed until the nonconformance is corrected. Samples that are analyzed prior to the resolution of a nonconforming event will be re-sampled, and/or reanalyzed once the corrective action has been initiated and is proven effective.

A copy of each closed nonconformance report shall be included in the quality assurance file and shall be maintained by the Project QA Officer. A template of nonconformance and corrective action report is provided in Appendix B.

11.1 FIELD CORRECTIVE ACTION

A corrective action shall be initiated during the field work when precision, accuracy, completeness, representativeness or comparability are not met or changes are made in the field that do not meet the scope of work requirements or other conditions are identified that are not consistent with the SAP. To document, a report shall be filed which lists the problems encountered and the corrective action implemented. A stop-work order may be issued by the Project QA Officer, if no resolution can be reached.

11.2 LABORATORY CORRECTIVE ACTION

If QA results for a particular analysis are outside the performance criteria described in this SAP or site-specific work plan (e.g., performance criteria for DQIs presented in Section 4) corrective action will be taken to ensure continued data quality. Corrective actions that may be taken include, but are not limited to:

- Rechecking calculations;
- Checking QC data on other samples;
- Auditing laboratory procedures;

- Repreparing and/or reanalyzing the sample if warranted;
- Accepting data with the acknowledged level of uncertainty; and
- Qualifying the data as unusable.

The laboratory QA Manager will be responsible for initiating laboratory corrective action within 48 hours of the time it was noted.

12 QUALITY ASSURANCE REPORTS TO MANAGEMENT

QA reports will be generated by Parsons and corresponding laboratories during the project. In addition, audit and performance evaluation reports will be submitted by auditors to management to ensure the quality of the project.

12.1 LABORORATORY QA REPORTS

The laboratory will summarize pertinent QA/QC issues in the laboratory data package case narrative report. These reports will include discussions of any conditions adverse or potentially adverse to quality, such as:

- Any laboratory or sample conditions which necessitate a departure from the methods or procedures specified in this plan,
- Any missed holding times or problems with laboratory QC acceptance criteria, and
- The associated corrective actions undertaken.

Such reports shall not prevent early notification to project management of such problems when timely notice can reduce the loss or potential loss of quality, time, effort, or expense.

12.2 FIELD QA REPORTS

Any field-related QA memorandums or forms shall be forwarded by field team leaders to the project manager, who will ensure that the project QA officer receives copies. The project technical director and project manager (or designated individual) will review these reports for completeness and the appropriateness of any corrective actions. The reports will be retained in the project files, and will be summarized in the QA report included in the final project documents. Appropriate steps will be taken to correct any QA/QC concerns as they are identified. The Parsons project manager will ensure that the technical project manager is informed of any significant QA/QC developments.

12.3 REPORTS OF AUDIT AND PERFORMANCE EVALUATION

As discussed in Section 9.3, audit reports will be written by auditors who have performed the audit within fifteen days after the completion of the audit. Serious deficiencies will be reported to the project manager within 24 hours to satisfactorily resolve the noncompliance in a specified and timely manner. The audit reports are directed to Parsons project manager, the Army, and the regulators (contact information see Section 3).

12.4 PROJECT QA REPORTS

A project QA report will be submitted after the project sampling and analysis is completed as part of the technical report. The QA report will summarize the overall QA information of the project, including information of laboratory performance, field performance, system performance, audit findings, and corrective actions. In addition, both validated data and laboratory and field QC data will be presented. The laboratory QA reports, field QA reports, project audit reports, and corrective

action reports will be used to assist in developing the final QA Report. The project QA report does not prevent internal QA memorandums or communications regarding QA issues.

The following elements, if applicable, will be addressed in the QA section or other section of the technical report:

- Project scope,
- Project description,
- Status of project,
- Sampling procedures (planned vs. implemented),
- Field quality control activities (planned vs. implemented),
- Analytical procedures,
- A summary of data usability assessments in terms of precision, accuracy, representativeness, completeness, comparability, and sensitivity,
- Any problems that could affect the quality of the data collected, the project schedule or the completion of the project,
- Changes in the project's experimental design, objectives, or staffing,
- The need for additional equipment to achieve project objectives, or any problems with equipment,
- Data presentation,
- Required corrective actions and effectiveness of corrective action implementation,
- Limitations on the use of measurement data generated, and
- Lessons learned

13 SAP REVISIONS AND DISTRIBUTION

This section presents procedures and requirements for SAP revisions (Section 13.1) and distribution (Section 13.2).

13.1 SAP REVISIONS

The generic SAP will be revised and updated every five years in accordance with USEPA (2004) requirement, or when there are changes warranted in response to project needs, or when directed by the approval authority. The project manager, QAO, and project chemist are responsible to determine if any changes to the SAP are warranted and their impacts to the quality of the project. If a change is desirable, the change will be incorporated into the site-specific work plans or issued as addendum to the generic SAP and approved by USEPA Region 2 and NYSDEC. Changes to the original SAP will only be implemented after the revision has been approved.

The quality assurance officer is responsible for revising the SAP. All project personnel should consult the QAO for the most recent approved version of the SAP.

13.2 SAP DISTRIBUTIONS

Table 19 lists all individuals who should get a copy of the approved SAP, either in hard copy or electronic format, as well as subsequent revisions: All the individuals identified in Table 19 will also receive all revisions, addenda, and amendments to the SAP. These individuals are responsible for removing all outdated material from circulation, distributing revised or added material to update any copies within their organizations.

All project personnel performing work related to sample collection, data producing, data assessment, data management, and data utilization should read the applicable sections of the SAP and perform the tasks as described. A project personnel sign-off sheet is presented in Appendix C and all identified personnel should read and sign off on the applicable sections of the SAP before beginning the tasks. Supervisory or oversight personnel are responsible for communicating the requirements of the applicable portions of the SAP to those doing work.

13.3 SAP ARCHIVING

The approved generic SAP and project-specific work plan, including reviewers' comments and responses to reviewers' comments will be archived in the appropriate project file. The files will be retained for the duration of the project or a minimum of five years, whichever is longer, or as dictated by project requirements (if longer than five years).

14 SPECIAL TRAINING/CERTIFICATION

Qualifications for quality assurance officer, field analyst, and data validators are specified in Section 3. In brief, field analyst should have: (1) completed a certification course or training by an experienced analyst who has demonstrated proficiency in the method; or, (2) demonstrated the proficiency by correlation of the analyst's results with laboratory confirmation analysis. Data validation will be performed by trained and experienced data validators. The lead validator will have at least two years experience and be familiar with USEPA Region 2 data validation requirements. The quality assurance officer should have the qualifications specified in the NYSDEC guidance.

Field sample collection team should be led by experienced engineer who has demonstrated proficiency in the sampling method.

Laboratory analyst should complete training by the laboratory and with qualifications deemed appropriate by the laboratory. The laboratories selected to perform analyses must be certified under the Environmental Laboratory Approval Program, implemented by the New York State Department of Health, and be capable of providing complete environmental analytical services consistent with USEPA protocols and NYSDEC ASP protocols.

Any other project specific special training should be recorded in the site-specific work plan.

15 DOCUMENTS AND RECORDS

All project documents (e.g., generic SAP, audit reports, internal QA/QC memorandums, interim progress reports, final reports) and records (e.g., field records and notes, communication logs) will be organized and kept consistent with the project management plan prepared by Parsons. All project documentation will be filed in the permanent project files. All project files will be maintained for the duration of the project or a minimum of five years, whichever is longer, or as dictated by project requirements (if longer than five years).

All the following files will be archived after the project is complete:

- Approved generic SAP and site specific SAP or work plan (including reviewers' comments, responses to reviewers' comments, addenda, and amendments),
- Sampling collection and handling records (e.g., field notebooks, operational record, global positioning system data, sampling instrument decontamination records, sampling instrument calibration logs, sampling location and sampling plan, drilling logs),
- Laboratory report (including chain-of-custody forms, sample receipt and tracking records including sample tags and shipping bills, case narrative, analytical log books, test method raw data and QC sample records, definitions of laboratory qualifiers, documentation of laboratory method deviations, and electronic data deliverables),
- Laboratory certification and QA manual,
- Computer documentation such as model input and output files as results of code and database test procedures,
- Audit reports/checklists, documentation of internal QA review, and corrective action reports,
- Interim progress reports and final reports,
- Billing receipts,
- Presentations to be made during and after the project,
- Communication logs, telephone logs,
- Documentation of deviation from methods,
- Data review reports, and
- Any other project related documents.

Electronic project files are maintained on a no-fault server and back-ups of project files on to magnetic tapes on the no-fault server are performed on a weekly basis and updated daily, Monday through Thursday.

Laboratory document control procedures shall be consistent with the NYSDEC ASP.

16 FIELD SAMPLING PLAN

16.1 INTRODUCTION

This section presents a generic Field Sampling Plan (FSP), which in specific terms, specifies the requirements and procedures for conducting field operations and investigations at Seneca Army Depot (or Depot). This generic FSP has been prepared to ensure (1) the data quality objectives specified for the Seneca Army Depot are met, (2) the field sampling protocols are documented and reviewed in a consistent manner, and (3) the data collected are scientifically valid and defensible. A site-specific work plan shall be prepared to supplement requirements and procedures for conducting site-specific field operations and investigations for each specific project or task, and shall reference this SAP document as appropriate to prevent repetition of information.

The National Contingency Plan specifies circumstances under which an FSP is necessary for Comprehensive Environmental Response, Compensation, and Liability Act response actions. For cleanup actions at the remedial investigation/feasibility study stage, the NCP requires lead agencies to develop sampling and analysis plans that provide a process for obtaining data of sufficient quality and quantity to satisfy data needs. Such sampling and analysis plans must include a field sampling plan. 40 CFR 300.430 (b)(8)(ii).

Guidelines followed in the preparation of this FSP are set out in the documents below:

- "Guidance for the Data Quality Objectives Process," (QA/G-4) (USEPA, EPA/600/R-96/055, August 2000).
- "Data Quality Objectives Process for Hazardous Waster Site Investigations," (QA/G-4HW) (USEPA, EPA/600/R-00/007, January 2000).
- "Guidance for Quality Assurance Project Plans," (QA/G-5) (USEPA, EPA/240/R-02/009, December 2002).
- "Guidance on Choosing a Sampling Design for Environmental Data Collection," (QA/G-5S), (USEPA, EPA/240/R-02/005, December2002).
- "Guidance for Preparing Standard Operating Procedures," (QA/G-6), (USEPA, EPA/240/B-001/004, March 2001).
- "Guidance for Data Quality Assessment: Practical Methods for Data Analysis," (QA/G-9), (USEPA, EPA/600/R-96/084, July 2000).
- "Data Quality Objectives Process for Hazardous Waster Site Investigations," (QA/G-4HW) (USEPA, EPA/600/R-00/007, January 2000).

All staff participating in SEDA activities, including remedial investigations, feasibility studies, and remedial projects are required to be familiar with this FSP. The FSP shall be in the possession of the field teams collecting the samples. All contractors and subcontractors shall be required to comply

with the procedures documented in this FSP in order to maintain comparability and representativeness of the collected and generated data.

Controlled distribution of the SAP (including the FSP presented in this section) will be implemented by Parsons to ensure the current approved version is being used. A distribution list is presented in Table 19. Persons listed in the distribution list will also receive revised version or addenda whenever revisions are made or addenda added to the SAP to assure (1) all parties holding a controlled copy of the SAP shall receive the revisions/addenda and (2) outdated material is removed from circulation. The document control system does not preclude making and using copies of the SAP; however, the holders of controlled copies are responsible for distributing additional material to update any copies within their organizations.

16.2 PROJECT SCOPE AND OBJECTIVES FOR FIELD SAMPLING ACTIVITY

This section presents the project scope and objectives for field sampling activity. The subsections present a summary of project data quality objectives, types of sample analysis, and field activities.

16.2.1 DATA QUALITY OBJECTIVES (DQOs)

Data Quality Objectives (DQOs) define the type, quantity, and quality of data that are needed to answer specific environmental questions and support environmental decisions. The development of DQOs for a specific site and measurement takes into account project needs, data uses and types and needs, and data collection. These factors determine whether the quality and quantity of data are adequate for its end use. DQOs are implemented so the data are legally and scientifically defensible. DQOs for this program are described in greater detail in Section 4 of the document.

16.2.2 SAMPLE ANALYSIS SUMMARY

The number and type of analyses will be determined on a per-task basis and will be specified in each site-specific work plan. Types and frequencies of QC samples (MS/MSD, trip blanks, equipment blanks, duplicates, etc.) required for all sampling activities are described in Section 4. Sample containers, preservatives, and holding time for soils/sediments and aqueous samples are provided in Tables 5-A and 5-B, respectively.

16.2.3 FIELD ACTIVITIES

Field activities associated with this project will include installation of system components (i.e., biowall and groundwater monitoring wells), soil sampling, baseline groundwater sampling, and subsequent process monitoring. These activities may include the following:

- Excavation;
- Soil boring sampling;
- Confirmation soil sampling;

- Stockpile sampling;
- Groundwater monitoring well installation;
- Groundwater monitoring well development;
- Water level measurements;
- Groundwater sampling;
- Field measurements of groundwater parameters;
- Soil sampling of biowall spoils;
- Sampling equipment decontamination;
- Aquifer testing; and
- Record keeping.

Field activities conducted at each site will be described in each SS-WP. Maps presenting planned field activities will be included in the SS-WP.

16.3 FIELD OPERATIONS

16.3.1 SOIL AND ROCK DESCRIPTION

16.3.1.1 Soil Description

Soils logged during test pit activities or recovered from soil and bedrock borings will be classified according to the Unified Soil Classification System (USCS), with descriptive text added to the USCS following the procedure outlined by Burmister.

Soil descriptions will be based on a clean view of the sidewall of a test pit or the clean face of split spoon sample that has been cut in half length-wise. The descriptions will be recorded on the Test Pit or Boring Report form (see Appendix G) using the following order and format:

- Color (while wet);
- Grain size;
- Major soil component descriptor (CLAY, SILT, SAND, GRAVEL, PEAT), with modifiers as applicable (micaceous, fibrous, etc.);
- Other components in decreasing order using the Burmister Method to quantify amounts (and, some, little, trace with + or - as applicable);
- Density from blow count data (if split spoon);
- Other descriptive modifiers such as stratification, plasticity, staining, minor minerals (if recognizable), unique materials or features, and odor (if present);
- Moisture content if the drilling method used does not interfere with the sample moisture and if the sample is above the water table;
- Possible origins will be given if enough information is available (i.e. fill, alluvium, till, glacio-marine, etc) on the next line after the soil description in parentheses;
- In some areas, the soil (clay, silt, sand, peat) may only be a minor component, the matrix to foreign debris. Note approximate percentages of debris vs. soil;
- General debris types (write in the “Remarks” section); and

- For test pits or trenches, sketch out cross sections of the excavation on the back of the log form.

The following is an example of an acceptable soil description:

> Light brown, coarse to fine micaceous SAND, some - Silt, little + coarse + Gravel, medium dense, well graded, weakly stratified showing grade bedded, minor iron-oxide stain on quartz grains, dry. (Alluvium)

16.3.1.2 Rock Description

Bedrock descriptions are dependent on the classification of the rock types present (igneous, sedimentary, or metamorphic). The rock materials retrieved during coring operations will be described on the Bedrock Core Form (Appendix G), as applicable, using the following parameters:

- Color - The overall color of the rock, not a particular mineral;
- Grain Size - The size of crystals or clasts making up the rock;
- Texture - This applies only to igneous and some metamorphic rocks, and pertains to whether the rock is crystalline or glassy, equigranular, or porphyric in nature;
- Major Minerals - Applies to the identifiable minerals present as necessary as modifiers to the rock type, i.e. mica Schist, feldspathic Granite, quartz-mica Gneiss;
- Rock Type - Granite, Gneiss, Amphibolite, Argillite, Sandstone, Limestone, Greywacke, etc;
- Bedding and/or Foliation - Describes lineations within the rock. i.e. massive, poorly foliated, well bedded, cross-bedded, etc. The description in the log will include at least an approximate angle of any foliation or bedding, if present;
- Continuity - Describes joints and fractures, or the lack of, in the rock, it may also describe cross-cutting veins of materials different from the primary rock type. Fracture, vein and joint angles will be referenced to the foliation. Approximations will be made based on core recovery, weathering, fracture density, etc., regarding the openness of any fracture or joint;
- Competence - Describe the weathering features of the rock. Weathering features, combined with rock type and continuity, will give the overall hardness of the rock; and
- Other - Describe secondary minerals, folding features, etc.

Both the overall core length as well as individual pieces of core (greater than 4 inches in length) will be measured. This data is used for the calculation of Rock Quality Designation (RQD) factors and for the interpretation of fracture spacing. Core recovery will be recorded in two manners: as the ratio of core recovered to length of core run; and, as a percentage recovery. i.e. 3.5 feet of 5.0 feet cored, 70%. The RQD will be calculated by: 1) summing the length of all the pieces greater than or equal to 4 inches in length recovered in the core barrel; and then, 2) by dividing this sum by the cored interval length. The resulting value is expressed as a percent and is recorded on the Bedrock Core Form (Appendix G).

16.3.2 SITE RECONNAISSANCE, PREPARATION, AND RESTORATION PROCEDURES

Areas designated for intrusive sampling shall be surveyed for the presence of underground utilities. Utility locations are determined using existing utility maps, and in the field, are verified using a hand-

held magnetometer or utility probe. Vehicle access routes to sampling locations shall be determined prior to any field activity.

A centralized decontamination area shall be provided for drilling rigs and equipment. The decontamination area shall be large enough to allow storage of cleaned equipment and materials prior to use, as well as to stage drums of decontamination waste. The decontamination area shall be lined with a heavy gauge plastic sheeting, and designed with a collection system to capture decontamination waters. Solid wastes shall be accumulated in 55-gallon drums and subsequently transported to a designated waste storage area. Smaller decontamination areas for personnel and portable equipment shall be provided as necessary. These locations shall include basins or tubs to capture decontamination fluids, which shall be transferred to a large accumulation tank as necessary. The designated areas of decontamination shall be specified in the SS-WP.

Parsons field office will be located in Building 125 at the Depot, unless otherwise specified in the SS-WP.

Each work site or sampling location shall be returned to its original condition when possible. Efforts shall 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, all drums, trash, and other waste shall be removed. Decontamination and/or purge water and soil cuttings shall be transported to the designated locations as described in Section 16.3.13.

16.3.3 GEOPHYSICAL SURVEYS

It is not expected that geophysical surveys will be required during activities at the SEDA. If they are required for a specific site, the SS-WP will include details of the process, including equipment, establishment of grid patterns, and QC procedures.

General requirements for all geophysical surveys are: (1) the subcontractor shall have a state licensed geologist or engineer to supervise AFCEE work, (2) the locations of boreholes logged with geophysical instruments shall be shown on a site map, (3) the locations of surface geophysical grid system layouts shall be shown on a site map, (4) the location of areas analyzed with subsurface geophysical techniques shall be shown on a site map (5) final results shall be presented in plan views and cross sections. Contours shall be used where appropriate, (6) the interpretation of results shall discuss positive and negative results as well as limitations of the method and data and, (7) the interpretation of the data shall be incorporated into the conceptual site model.

16.3.4 SOIL GAS SURVEYS

Soil gas surveys are anticipated in the SEDA program. If this changes for a specific site, soil gas survey methods and QC procedures will be included in the SS-WP.

The primary function of soil gas surveys is to assist in identifying potential source areas for soil and groundwater contamination. Soil gas shall also be used in small source areas to help target soil boring, monitor well, and indoor air sampling locations. Soil gas sampling networks shall be designed to obtain all necessary information with a minimal expenditure of time and resources. The development of the sampling network shall be based on background information, properties of the vadose zone, and hydrogeologic properties of the area. Soil gas sampling procedures are described in Section 16.4.1.7.

Common sampling schemes include grids, transect lines, biased, random, and combinations. Grids consist of sampling points on perpendicular lines at equal distances along the lines. The size of the grid shall be dependent upon site characteristics and sampling objectives. The transect line sampling network is typically used to find a source area of contamination. Sampling points are placed along a line between the area of impact and the suspected source area. In a biased sampling network, sample points are placed near the suspected source of contamination to locate "hot spots" and further delineate the extent of contamination. This sampling network shall not be used for unknown conditions. Random sampling networks use a numbered grid system. The sample points are selected by a random number generator. This network is typically used in areas where little information is available or no contamination is suspected. Combined type of sampling network, consists of a combination of the above reference networks. The interval between sampling points shall be dependent on the objectives of the investigation.

The type(s) of sampling schemes selected shall be dependent on site conditions and the data quality objectives for the project. Soil gas sampling shall be used when groundwater sampling indicates contamination or when vadose zone contamination is suspected.

16.3.5 SOIL BORING ADVANCEMENT

Soil borings advanced during the project shall be performed using hollow-stem auger (HSA) technology. Hollow stem augers, 4.25 or 6.25 inch inner diameter (ID) (when coring), will be used to drill each boring. The borings will be advanced to "refusal" which will represent the depth of the "competent" bedrock. Auger "refusal" in "competent" shale will be defined as the depth (after penetrating the weathered shale) when augering becomes significantly more difficult and auger advancement is slow. Samples shall be collected in accordance with procedures outlined in Section 16.4 of this SAP.

After the boring is completed, and if a well is not to be installed, it shall be refilled to the ground surface with lean grout containing at least 3% bentonite powder by volume. The cement/bentonite grout seal shall be placed from the bottom of the boring to approximately 3 feet below ground surface by pouring the mixture into the hole. The grout mixture shall consist of Portland cement (ASTM C 150-86) and water in the proportion of not more than 7.0 to 8.0 gallons (gal) of clean water per bag of cement [1 cubic foot (ft^3) or 94 pounds (lb)]. Additionally, 3 percent by weight of bentonite powder will be added to help reduce shrinkage of the grout mixture. The grout will be allowed to set a minimum of 48 hours. If the borehole is greater than 15 feet and groundwater is present in the borehole, the grout will be pumped through a tremie pipe to the bottom of the boring. Grout will be pumped in until undiluted grout discharges from the bore hole at the ground surface. A bentonite backfill consisting of bentonite pellets will be placed from the top of the cement/bentonite grout seal to the ground surface and allowed to hydrate.

16.3.6 GROUNDWATER WELL INSTALLATION

16.3.6.1 Unconsolidated Monitoring Wells

Borings will be advanced as described in Section 16.3.5. Installation of overburden monitoring wells will begin as soon as possible following the completion of the boring. Once installation of the well has

begun, the installation process will be continuous until the well has been grouted and all augers or casings have been removed.

The Field Inspector will measure, or observe the driller measure, the depth to the bottom of the borehole and will confirm that the depth is sufficient to proceed with well installation. Prior to placement in the hole, all PVC well materials will be steam cleaned. The well assembly will be lowered into the borehole, with additional sections attached as the well is lowered into the hole. All threaded connections will be threaded "hand-tight" and sealed before each section is lowered into the hole. No tools will be used to tighten PVC joints, as over-tightening may result in breakage.

Once the well assembly has reached the bottom of the borehole, an expansion plug will be installed in the top of the PVC to ensure that nothing is dropped in the well. The well assembly will be raised 0.1 to 0.3 feet off the bottom of the borehole. Next, a volume of sand equivalent to 1 to 2 feet of borehole annular space will be poured into relatively shallow wells; in the case of wells greater than 15 feet deep, a tremie pipe will be used to place the sand pack. Following the addition of the sand, the PVC well assembly will be raised an additional 0.1 to 0.5 ft to provide a sand cushion at the well point. Filter sand will be added in 1 to 3 foot increments while the augers or casing are removed. When the filter sand is 0.5 to 1-foot above the top of the screen, a finer grained sand pack material, 6 inches thick, will be placed at the top of the filter sand. The fine sand will serve as a barrier between the filter sand pack and the bentonite seal to prevent infiltration of the bentonite into the sand pack around the well screen

The bentonite seal will be placed above the fine sand pack, and should be at least 1 foot thick on shallow wells and up to 2 feet thick on deeper wells. Prior to grouting the remainder of the hole, the seal will be allowed to set for an hour. After the seal has set, the hole will be grouted to 3 feet below the ground surface using 3% by weight bentonite to cement. All grout seals installed below the water table will be tremied into place. Installed grout seals will be allowed to settle and set for 24 hours prior to being inspected. Additional grout will be added to bring the grout level to approximately 3.5 feet below ground surface for surface well completion. The spoils associated with grouting operations will be handled as a waste stream, and the surface completion for the well will be completed two to seven days after well installation. If required, the boring will be logged according to the procedures in Section 16.3.1, and samples may be taken according to the procedures outlined in Section 16.4. All details of the well completion activities will be recorded on the Well Completion form (Appendix G).

16.3.6.2 Bedrock Wells

Bedrock well installation will be identical to the unconsolidated well installation with the exception of the bedrock coring procedures and installation of a steel casing across the bedrock/overburden boundary.

After initial drill refusal at the top of bedrock, the hole will be drilled or reamed 3 to 4 feet into competent bedrock to confirm the bedrock surface and allow for the installation of the outer steel casing into the competent bedrock. Following the initial coring into bedrock, approximately 2 feet of bentonite chips will be added to the bottom of the borehole and allowed to hydrate for 15 minutes. The steam-cleaned steel casing will be advanced to 2 feet above the bentonite seal, and grout will be tremied into the hole until undiluted grout flows from the top of the hole. The casing will be pushed

into the bentonite seal. If the grout level drops during this operation, more will be added to the hole until the level stabilizes 3 feet below the ground surface.

The grouted casing will be allowed to set for a period of 48 hours prior to the initiation of coring operations, after which the boring will be advanced to a maximum depth of 20 feet below the steel casing using HQ size core and core barrel. During coring, a potable analyte-free water will be pumped into the borehole to serve as a lubricant and to remove the fine rock flour and shale chips from the hole. The water will be recirculated into the hole after passing through a steel bath with several baffles to contain most of the rock flour and shale chips, preventing them from being reintroduced into the borehole. A description of the rock core will be recorded on the Bedrock Core Log according to the procedures outlined in Section 16.3.1.2, and the Well Completion will be detailed on the Bedrock Well Completion form. Both forms are contained in Appendix G.

16.3.7 MONITOR WELL DEVELOPMENT

All installed groundwater monitoring wells require development prior to sampling. Development will be performed to remove sediment from inside the well casing and to flush fine materials from the portion of the formation adjacent to the screen. Development will be accomplished using a submersible pump and will be continued until a minimum 3 well volumes of water have been removed from the well and until pH, temperature, specific conductance, dissolved oxygen (DO), and turbidity stabilize. Stabilization will be defined as three consecutive readings taken at 3 minute intervals within 10% of each other. If the water remains turbid, development will continue until the turbidity of the water produced has been stable after the removal of several additional casing volumes.

A development record will be maintained for each monitoring well. The development record will be completed in the field by the Parsons representative. Development records will include:

- Groundwater monitoring well number;
- Date and time of development;
- Development method;
- Predevelopment water level and well depth;
- Volume of water produced;
- Description of water produced; and
- Post development water level and well depth.

The Well Development field form is contained in Appendix G.

16.3.8 ABANDONING MONITOR WELLS

No monitoring wells are scheduled to be abandoned as part of this remedial project. If this changes, an addendum to this FSP will be added that describes specific well abandonment procedures.

All abandonment of monitor wells directed by AFCEE shall be performed in accordance with federal, state and local laws and regulations. The well should be cleared of all obstructions prior to abandonment. Obstructions such as pumps, pipes, wiring, and air lines must be pulled. An attempt should be made to pull the casing when it will not jeopardize the integrity of the borehole. Before the

casing is pulled, the well should be grouted to near the bottom of the casing. This will provide a seal if the well collapses after the casing is pulled.

If slurry is used to seal the well, a mud balance and/or Marsh Funnel shall be used to ensure that the density (lbs/gal) of the abandonment mud mixture conforms to the manufacturer's specification. All abandoned monitor wells shall be checked 24 to 48 hours after mud/solid bentonite emplacement to determine whether curing is occurring properly. More specific curing requirements or quality assurance checks may be recommended by the manufacturer and shall be followed. Additionally, if significant settling has occurred, a sufficient amount of mud/solid bentonite shall be added to attain the initial level. These slurry/solid bentonite curing checks and any addition of mud/solid bentonite shall be recorded in the field logs.

Copies of all completed field logs should be submitted to the AFCEE COR. Additionally, the ERPIMS well records for all wells abandoned should be modified to annotate abandonment and the updated electronic well records submitted to AFCEE/MSC.

16.3.9 AQUIFER TESTS

16.3.9.1 Aquifer Testing For Hydraulic Properties

16.3.9.1.1 General

Equipment shall be decontaminated and water levels measured according to the specifications of Section 16.3.12. The contractor shall demonstrate that the assumptions of the selected analytical methods for deriving the hydraulic properties match the hydrogeological conceptual site model, and meet the DQOs.

16.3.9.1.2 Slug Tests

Slug tests are applicable to rocks or unconsolidated deposits of low to moderate hydraulic conductivity. Testing of several wells is necessary to characterize an aquifer because slug tests only measure aquifer properties immediately adjacent to the borehole or well. The water level shall be static before the test begins. That is, it must not be recovering or receding as a result of sampling, development, pumping of nearby wells, or related activities. The test shall be performed using a slug or by withdrawing water from the well. No fluid shall be put into the well.

When designing a slug test, the geologist should keep in mind the following criteria: (1) volume of the slug, (2) diameter of the well, (3) depth and length of the screened interval, (4) method and frequency of water level measurements, (5) barometric pressure and, (6) the method used to analyze the data. If the static water level is below the top of the screen or open section of the well, a falling-head test should not be performed. The slug test shall continue until the water level has recovered to at least 80 percent of its static (pretest) level.

All valid water-level or drawdown versus time data resulting from these tests should be appended to the draft and final reports describing the analysis of these tests. These field data should be provided in ASCII electronic format. Additionally, these field data and the calculated hydraulic conductivity values should be loaded into ERPIMS and submitted to AFCEE/MSC.

16.3.9.1.3 Pumping Tests

The contractor shall use monitor wells as observation wells as much as possible. The pumping rate shall be determined by conducting step-drawdown tests prior to the pumping test. The well shall be pumped at predetermined rates in order to determine the optimum pumping rate. If a lower pumping rate is preferable because of factors such as nearby supply wells, areas with floating product, disposal costs, or limited storage facilities, the lower rate shall be approved by AFCEE. In addition, barometric pressures should be monitored at the beginning and, at a minimum, at the end of the test to evaluate the impact barometric pressure may have on the test. The test shall not begin until water levels in all wells have completely recovered. The contractor shall monitor and regulate the discharge valve for either a constant-discharge or constant-head test. The discharge rate shall be measured at least ten times during the first 100 minutes of the test and at least every time water levels are measured thereafter. Discharge rates shall be measured in accordance with Section 16.5.4.3. Water levels shall be measured at least ten times per log cycle for the first 100 minutes of the test and at least once every hour thereafter. The pumped water shall be disposed of so as not to recharge the portion of the aquifer being tested or otherwise affect the validity of the test. Time-drawdown or distance-drawdown data shall be analyzed during the test. The test shall be terminated when collection of additional data does not affect results (e.g., when water levels are essentially at equilibrium, or when a well in a zone with low hydraulic conductivity does not yield sufficient water to continue). Test durations may range from two hours to a week or more. A common test period is 24 hours.

All valid water-level or drawdown versus time data resulting from these tests should be appended to the draft and final reports describing the analysis of these tests. These field data should be in ASCII electronic format. Additionally, these field data and the calculated hydraulic parameter values should be loaded into ERPIMS and submitted to AFCEE/MSC.

16.3.9.1.4 Other Test Methods

The aquifer hydraulic parameters can be estimated from well specific capacity and from step-drawdown tests. For low hydraulic conductivity rocks, ASTM D-4630 or D-4631 is applicable. For clay, ASTM D-1587 and D-2434 are applicable.

16.3.10 TEST PIT EXCAVATION

The primary objective of test pitting is to provide a means for the visual evaluation of subsurface soils and the collection of soil samples or to investigate anomalies discovered during the geophysical surveys. Test pits and trenches shall be excavated by hand or by power equipment to permit detailed observation of in-situ materials. Hand digging around specific materials encountered may be necessary to prevent puncture or damage of the objects. Sufficient space should be maintained between trenches/pits for the placement of soil stockpiled for cover as well as to allow access and free movement by support vehicles and operating equipment.

During field operations, the locations of all proposed test pits shall be marked out prior to the initiation of excavation. While excavating in parking areas, improved grassy areas, or areas that may be contaminated, excavated materials will be placed on polyethylene sheets beside the test pit. While excavating in landfill areas with unimproved dirt surfaces, only obviously contaminated materials, different from surface material, shall be placed on plastic. The staging area should include run-off containment features, and the top 6 to 12 inches of soil will be kept separate from the deeper soil so

that it can be used as cover material when the test pit is backfilled. The size and depth of the test pit or trench will be described in the Work Plan.

When appropriate, air sampling will be performed during test pitting to support two broad-based directives: the protection of workers and the protection of public safety. Air sampling shall be performed using a PID with an 11.7 mV lamp to monitor for volatile organic compounds (VOCs) in the air. It will be used to monitor air in the breathing space of workers during test pit activities. The procedures for any necessary air monitoring will be specified in the Site Specific Health and Safety Plan (SS-HSP).

The field geologist will keep detailed records on the Test Pit Report Form (Appendix G), which includes the following information:

- soil descriptions and stratigraphic changes;
- relative soil moisture;
- depth to groundwater (if encountered);
- visible signs of staining (natural or otherwise);
- results, type, and time of monitoring measurements;
- sampling locations, depths, and time;
- time of excavation;
- reason for terminating excavation; and
- type of excavator.

At no time will any personnel be permitted to enter the excavation area. Any excavated containers filled with liquid or solid substances will be overpacked and tested for hazardous constituents. If unexploded ordnance (UXO) or explosives are observed in excavated soils where they were not anticipated, the excavation will be stopped until qualified UXO personnel can examine the situation and recommend a course of action to the Safety Officer.

The test pit will be closed by backfilling the pit with the soil removed from it. As discussed above, the surface soils will be backfilled last. If the pit is not to be closed immediately after the required samples have been obtained, the excavation will be barricaded to prevent accidental entry by personnel working on the site. Each excavation will be marked after closure, as necessary, for identification of the location.

16.3.11 SURVEYING

The locations and elevations of monitoring wells, soil borings, surface soil samples, sediment samples and surface water samples will be surveyed by a surveyor registered in the State of New York. The elevation of the ground surface adjacent to each surveyed point and measurement datum will be measured relative to an existing benchmark location referencing the Base grid system. Survey of the new wells will take place as follows:

- Horizontal locations for monitoring wells, soil borings, surface soil, sediment and surface water sample locations will be measured relative to Northing and Easting in State Planar Coordinates, NAD 1983, accuracy ± 0.1 feet.
- The elevation of the ground surface adjacent to each monitoring well will be measured relative to NAVD 1988, accuracy ± 0.1 feet at stake or pin in collar.

- The elevation of the top of the well protective casing and top in the well casing will be measured relative to NAVD 1988, accuracy ± 0.1.

Monitoring wells will have three elevations with varying levels of accuracy; the first for the top of well's PVC inner casing at a notch placed by the surveyor, a second for the top of the well's protective outer casing at the crown of the cap, and the last for the elevation at a pin placed in the collar of the well at the ground.

All monitoring wells shall be resurveyed at a minimum every five years, with the approval of AFCEE.

16.3.12 EQUIPMENT DECONTAMINATION

The following decontamination procedure will be followed for equipment to be used for collecting samples for analytical testing:

- Scrub with laboratory-grade detergent (Alconox);
- Rinse with copious quantities distilled water;
- Air dry;
- Wrap in aluminum foil (if being re-used)

Precautions will be taken to minimize any impact to the surrounding area that might result from decontamination operations, and any deviations from these procedures will be documented in the field notebook and on the appropriate sampling record.

Laboratory-supplied sample containers will be cleaned and sealed by the laboratory. The type of container provided and the method of container decontamination will be documented in the laboratory's permanent record of the sampling event.

16.3.13 INVESTIGATION DERIVED WASTE (IDW) DISPOSAL

Decontamination Fluids. Decontamination fluids will be collected in DOT-approved 55-gallon drums. The drums will be labeled as investigation derived wastewater and temporarily stored in a secured area to be determined prior to commencement of field activities. The drums will be stored on wooden pallets in a plastic-lined containment area or in other approved secondary containment structures pending characterization and disposal.

Drill Cuttings. Drill cuttings/test boring soils will be contained in 55-gallon drums. The soils will be segregated by drill location as is practical. The drums will be labeled as investigation derived waste soils from the corresponding boring or source area and temporarily stored in a secured area to be determined prior to commencement of field activities. The drums will be stored on wooden pallets in a plastic-lined containment area or in other approved secondary containment structures pending characterization and disposal.

Development and Purge Water. All development and purge water will be contained in 55-gallon drums. The drums will be labeled as investigation derived wastewater and temporarily stored in a secured area to be determined prior to commencement of field activities. The drums will be stored on

wooden pallets in a plastic-lined containment area or in other approved secondary containment structures pending characterization and disposal.

For IDW waste other than soil or water such as decontamination fluids and personal protection equipment, the disposal evaluation involved the following steps. Under RCRA, wastes are classified as hazardous if they are listed wastes or characteristic wastes. Waste specific information, such as manifests, bills of lading, storage records or records of waste sources must be used to document that a waste is a RCRA-listed waste; otherwise, in the absence of any other information, the waste in question cannot be considered a listed waste. Drummed cuttings, PPE, or purge water generally are not considered listed hazardous wastes since these material produced at the Seneca sites generally do not meet any of the regulatory definitions described in 40 CFR 261, (i.e. F-, K-, P- or U- listed wastes). The only listed waste generated during the investigation program is waste that contained methanol. Methanol was used in the decontamination process (per EPA direction), which makes the decontamination fluids an F003 listed hazardous waste. An F-listed waste classification refers to non-specific hazardous waste sources that contain methanol as a component of a spent solvent mixture. In order to limit the generation of hazardous waste due to the derived from and the mixture rules for listed wastes, methanol should not be mixed with soils or other liquids. Additionally, during the decontamination process, washable rubber bibs will be worn to prevent contamination of disposable PPE. Therefore, the disposable PPE is not a hazardous waste based upon the derived from or mixture rule and will be disposed of as uncontaminated refuse.

16.3.14 CORRECTIVE ACTION

A corrective action shall be initiated during the field work when changes are made in the field that do not meet the scope of work requirements or other conditions are identified that are not consistent with the SAP. Section 11.1 of the generic SAP describes corrective action procedures for field activities.

16.4 ENVIRONMENTAL SAMPLING

16.4.1 SAMPLING PROCEDURES

The construction material (e.g., plastic, PVC, metal) of the sampling devices described below shall be appropriate for the contaminant of concern and shall not interfere with the chemical analyses being performed.

All purging and sampling equipment shall be decontaminated according to the specifications in Section 16.3.12 of this SAP prior to any sampling activities and shall be protected from contamination until ready for use.

16.4.1.1 Water Level Measurement

Prior to the initiation of sample collection, static water levels will be measured for all of the wells at the site. An electric water-level probe will be used to measure the depth to groundwater below the datum to the nearest 0.01 foot. The datum is usually the top of the well's PVC casing, where a notch has been cut or a permanent black mark has been made at the measuring point. The exact datum location is especially important if the casing has been cut off at an angle. A Groundwater Elevation form is included in Appendix G.

16.4.1.2 Groundwater Sampling

Groundwater sampling for monitoring wells and microwells will be performed according to the Draft SOP titled Groundwater Sampling Procedure, Low Flow Pump Purging and Sampling (USEPA, May 15, 1995) and the comments from the EPA dated May 10, 1996. Low flow methods will be used due to past high turbidities in the purge and sampling water in monitoring wells at SEDA. The pumps used on the project will be low flow centrifugal or bladder pumps constructed of stainless steel or Teflon; the tubing used will also be Teflon, and each well will have its own dedicated tubing.

A polyethylene ground cloth and 5-gallon bucket will be placed beneath all sampling equipment during well purging and sampling to prevent the spread of contaminated groundwater, and if a gas-powered generator is used to drive the pump motor or controller, the generator must be placed a minimum of 25 feet downwind of the well to limit the incidence of cross-contamination during sampling. Pump, safety cable, tubing and electrical lines will be lowered slowly into the well to a depth corresponding to the center of the saturated screen section of the well. The pump intake will be set at the center of the saturated screen section of the well.

The water level will be measured again, with the pump in the well, before starting the pump. Pumping will be performed at a rate of 200 to 500 milliliters per minute, as allowed by the recharge rate in the well. Ideally, the pump rate should cause little or no water level drawdown in the well; and, if necessary, pumping rates will be reduced to the minimum capabilities of the pump to avoid pumping the well dry and/or to ensure stabilization of indicator parameters. These parameters, turbidity, temperature, specific conductivity, pH, dissolved oxygen (DO), and oxidation-reduction potential (ORP), will be measured continuously using calibrated instruments (see Section 6). It is anticipated that the instruments used will be the Lamotte 2020 Turbidity Meter and the Horiba U-22. Readings will be logged by field personnel at approximately 3 minute intervals. The well will be considered stabilized and ready for sample collection when three successive readings remain within the following criteria:

- $\pm$ 0.05 for pH;
- $\pm$ 3% for conductivity;
- $\pm$ 10% for temperature, DO, ORP; and
- $\pm$ 5 NTUs for turbidity with the turbidity below 10 NTU.

The flow-through cell will be removed upon well stabilization, and sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection. In some very low-yielding formations it may not be possible to sample with minimal drawdown even using low pumping rates. It should be noted that if the water level will not stabilize at minimum pumping rates and the water level is drawn down below the top of the pump, then stabilization of the indicator parameters may not be possible. In the past, these wells have been pumped to dryness and sampled as soon as they recovered sufficiently. Approval to sample in this manner will be required from the project manager, task manager, or site manager.

After purging the well, the sampling team will change to new outer gloves for sample collection. Groundwater samples for volatile analyses will be collected first, before any of the other parameters of interest, and will be obtained in a manner that will minimize the loss of volatile compounds. VOC samples will be collected directly into pre-preserved sample containers. All sample containers should be

filled by allowing the pump discharge to flow gently down to the inside of the container with minimal turbulence. The sampling flow rate for volatiles should be accomplished with a gradual reduction in the flow rate down to approximately 100 milliliters per minute and sustained hydraulic head pressure within the sampling tube to reduce aeration, bubble formation, turbulent filling of sample bottles, and loss of volatiles due to extended residence time in the tubing. The sample discharge for all other analytical parameters can be a continuous flow of up to 500 milliliters per minute.

Groundwater samples will be collected with the required quality assurance/quality control samples, then transmitted to the laboratory for chemical analysis in accordance with the Chemical Data Acquisition Plan (CDAP). Samples will be preserved and stored in an ice-filled cooler immediately after sampling is complete. Data regarding groundwater sample collection will be recorded on the Groundwater Sampling Record (Appendix G). Chain-of-Custody records will be maintained as described in Section 16.4.3.

16.4.1.3 Soil Boring Sampling

During drilling operations, soil samples will be collected continuously using a standard three-inch diameter, two-foot long carbon steel split spoon barrel. Split spoons of 2-inch diameter may be used only if the same interval will not be selected for chemical analysis. Immediately after the opening of a split spoon sampler the contents of the sample will be screened for VOCs and, if necessary, radiation. One to three readings will be taken along the sample with additional readings taken if additional distinctive zones are observed. These data will be recorded in the appropriate locations on the Boring Log Form (Appendix G). Descriptions of the sample will also be entered on the Boring Log Form.

Typically, three samples from each boring will be selected for chemical analysis: 1) 0 to 12 inches below grade; 2) immediately above the water table; and 3) between samples (1) and (2). The intermediate sample will be collected at a depth where one of the following site specific items occurs: (1) a stratigraphic change such as the base of the fill, (2) evidence of perched water table, (3) elevated photoionization detection (PID) readings, or (4) visibly affected soil (e.g., oil stains). If none of these occur, then the intermediate sample will be collected at the halfway point between the samples collected at the surface and at the water table. If intermediate split spoon samples exhibit elevated PID readings, the one with the highest concentration will be chosen as the intermediate sample.

Samples to be analyzed for volatile organic compounds will be collected first in two 40 ml vials with septum seals; these soil samples will not be homogenized or composited during the sampling process. All VOA sample bottles will be completely filled, leaving no void space. The remaining soil from the spoon will be mixed (homogenized) in a decontaminated stainless steel bowl with a decontaminated stainless steel utensil and placed in appropriate sample containers.

Sampling information will be recorded on the Soil Sampling Report (Appendix G). This form includes information such as the sample location, number, depth, time, description, and laboratory QA/QC sample names.

16.4.1.4 Surface Soil Sampling

Surface soil samples shall be collected by filling a bowl with soil from zero to two inches below any organic layer at the sampling location. After the soil to be sampled has been placed in the bowl, as much organic matter (roots, leaves, worms, etc.) as possible shall be removed from the bowl. Care will be taken to ensure that the soil placed in the bowl is not agitated extensively during this process if volatile

organic analysis samples are necessary. If VOA samples are necessary, they shall be collected first. All VOA sample bottles will be completely filled, leaving no void space. Following the collection of any VOA samples, the remaining soil shall be homogenized by mixing, and the rest of the necessary samples will be collected. Collected samples will be stored in a chilled cooler until they can be sent to the laboratory for analysis.

Sampling information shall be recorded on the Sampling Report form for soil/sediment (Appendix G). This sheet includes information such as the sample location, number, depth, time, Burmister description, and laboratory QA/QC sample names.

16.4.1.5 Surface Water Sampling

Prior to sampling at any surface water/sediment location, the direction of actual surface water flow directions shall be noted and recorded on a site map. The flow direction shall also be compared to the flow directions expected at the site to ensure that the samples planned for the downstream direction are truly at a downstream location. Typically, one background surface water/sediment sample will be collected from a location upstream of the assumed contaminant location. The location of this sample will be adjusted according to site flow conditions.

Sampling will begin at the most downstream location and progress upstream to ensure sampling activities at one location will not affect samples collected at another location. Surface water samples will be collected before sediment samples for the same reason. Before the surface water samples are collected at each location, measurements of temperature, pH, specific conductance, and ORP shall be taken by direct immersion of instrument probes (see Section 6) into the water body. If direct measurement is not possible, these measurements shall be taken from water collected and placed in a field container. If this procedure is followed, the water used to analyze field parameters shall not be used as the sample water; another sample will be collected.

Whenever possible, surface water samples will be collected from the surface water body by submerging a sample bottle into the water and angling the bottle at a 45-degree angle upstream to allow the bottle to fill without collecting any surface debris. If direct access to the water is not possible, decontaminated sampling equipment, such as a bailer, will be lowered into the water and the sample will be poured into the bottle. All pertinent field data will be recorded on the Surface Water Sampling Record (Appendix G), including distance from shore, water depth, color, and relative water velocity. The samples will be packed in an ice-filled cooler immediately after sampling.

16.4.1.6 Sediment Sampling

Sediment sampling will directly follow surface water sampling at each location. The sample shall be collected below any organic layer and up to 6 inches into the sediments and will be placed into a clean bowl prior to placement in sampling containers. If volatile organic analysis samples are required at the site, these shall be collected first, prior to any mixing of the sediment. The rest of the samples will be collected after the remaining sediments in the bowl have been stirred for homogenization purposes. All pertinent information shall be recorded on the Sampling Record form for sediment (Appendix G), including location, sample number, water depth, depth range over which the sample was collected, and a description of the sediment. The description will be recorded according to the procedures outlined in Section 16.3.1.1. Sediment samples shall be stored in an ice-filled cooler immediately after sampling is complete.

16.4.1.7 Soil Gas Sampling

Soil gas may be sampled using commercially available soil gas sampling probes, a gas tight syringe or bulb, a SUMMA® canister, sorbent tubes, or a Tedlar® bag.

When soil gas samples are collected using commercially available soil gas sampling probes, the probes are connected to a steel drive shaft used to push the probe to the desired sampling depth. The sampling container shall be a glass or metal bulb equipped with an entrance and exit spigot. The Tygon® tubing from the sampling probe shall be attached to the entrance spigot, and a second length of tubing shall run from the exit spigot of the bulb to a portable vacuum pump.

At each sample location, the sampling probes shall be driven to a previously determined depth of between 5 to 10 feet below ground surface.

When the probe is at the desired depth, the steel drive shaft shall be pulled back slightly, exposing the gas intakes on the sample probe. The vacuum pump shall then be switched on, drawing the gas contained in the interstitial spaces of the soil through the probe, tubing, and sample container. When 2 liters of gas have been drawn, the Tygon® tubing shall be clamped shut on the downstream side of the bulb (toward the pump) and then the upstream side of the bulb. The vacuum pump shall then be switched off. The volume of 2 liters shall ensure that the gas in the glass bulb originated from the soil interstitial space, rather than the tubing, so long as a reasonably short tubing length is used. Following sample collection, the sample container shall be labeled and the sample number recorded in the field log book along with the following information: soil gas sample or probe depth, apparent moisture content (dry, moist, saturated) of the sampled zone, if available, soil gas purge rate, sampling duration, sampling system leak rate, and pump vacuum, description of sample containers, location of sample analysis, location and grid layout of sampling stations.

Gas tight syringe or bulb samples are collected for on-site laboratory analyses. To collect a syringe sample, a fitting with a Teflon® septum shall be installed in the sampling line ahead of the purge pump. After purging the required volume, samples are collected. Bulb samples are collected using a manifold configuration.

SUMMA® Canister samples are collected for off-site laboratory analyses. A fitting for attaching the canister shall be installed in the sampling line ahead of the purge pump. Prior to sampling, the initial canister vacuum is measured, the canister is attached to the sample line, and the probe, etc. is purged. The canister sample is then collected.

Sorbent tubes may be used to collect samples for real-time field analysis (i.e., colorimetric tubes such as Draeger tubes) or for off-site laboratory analyses. The well or probe is purged, the sorbent tube is installed in the sampling line, and the required volume of soil gas is drawn through the tube. Colorimetric tubes are read directly, while sorbent tubes are capped and stored on ice (dry ice may be required) until being shipped to the laboratory.

Tedlar® bag samples can be collected for field analysis using real-time instruments or for off-site laboratory analysis. An oilless diaphragm pump is attached to the sampling line and a Tedlar® bag is attached to the pump exhaust. Samples shall be kept out of direct light and analyzed within 24 hours of collection to minimize the potential for loss, reaction, or degradation of VOCs. If Tedlar® bags are

used, a blank bag sample should also be collected for each lot of bags used. The blank bag sample should be filled with clean (ambient) air and submitted as a field blank. .

In addition to the information listed in Section 16.6, the following information shall be recorded. If only qualitative data are required, only items 1 and 6 are needed: (1) soil gas sample or probe depth, (2) apparent moisture content (dry, moist, saturated) of the sampled zone, (3) soil gas purge rate, sampling duration, sampling system leak rate, and pump vacuum, (4) description of sample containers (if any), (5) location of sample analysis, (6) location and grid layout of sampling stations, (7) instrument calibration.

16.4.1.8 Biowall Spoils Soil Sampling

Soil samples for VOC concentrations will be collected from biowall spoils to confirm the results of the preliminary waste characterization and the appropriate disposal threshold as established in the SS-WP. One discrete soil sample will be collected for every 25 to 300 linear feet of biowall spoils. Excavated soil spoils recovered during the continuous trenching operation will be stockpiled on plastic adjacent to the biowall, corresponding to the location the spoils originated. The discrete soil sample will be collected from the appropriate length of biowall that corresponds to at the highest screening concentration detected by a PID. The sampling frequency will be summarized in the SS-WP. This discrete sample will be analyzed for total VOCs via USEPA Method 8260. The soil sample will be placed in an iced cooler and submitted for analytical analysis by SW8260B on a one week turn around. Types of sample containers, sample volumes, and methods of preservation are identified in Table 5-A. Additional QA/QC samples will include daily trip blank/field blanks. The soil spoils will be disposed of in accordance with the work plan.

16.4.1.9 Composite Sampling

Occasionally, samples will be composited prior to chemical or physical characterization. Composites will be collected in the following steps:

- Discrete subsamples of equivalent size (weight, volume) will be collected from each of the selected locations and combined in a common receptacle;
- All necessary sample preparative operations (e.g., sample filtration, sleeve screening) will be performed on the subsamples in the receptacle;
- The material remaining after preparation will then be fully homogenized, generally by mixing;
- Any necessary preservatives will be added;
- The required samples will be collected and placed into clean sample bottles and packaged for shipment;
- Samples collected for certain types of analyses (volatile organic compounds and sulfides) WILL NOT be composited unless compositing is specifically requested by a regulatory agency.

16.4.2 SAMPLE HANDLING

16.4.2.1 Sample Volumes, Container Types, and Preservation Requirements

Types of sample containers, sample volumes, and methods of preservation are identified in Tables 5A and 5B. The laboratory will supply sample containers and preservatives in accordance with their own analytical procedures. A separate container may not be required for each parameter. The laboratory will add any necessary chemical preservatives prior to shipping the sample containers to the field.

16.4.2.2 Sample Packaging and Delivery

Samples will be delivered by common carrier to the designated laboratory for analysis daily or every other day, as required. The field team leader (or designee) will contact the laboratory to inform them of sample delivery before samples are to be picked up or delivered to the common carrier. The samples will be delivered in ice chests to the common carrier for overnight delivery. The chain-of-custody forms will be sealed in a plastic bag and taped to the inside lid of the chest. The chest will be sealed with custody seals and tamper-resistant tape, and the custody seals will be signed and dated by the sample custodian.

16.4.2.3 Sample Identification

Subsurface soil borings will be numbered consecutively beginning with SB-01 (soil borings) or MW-1 (monitoring well borings) or starting with the next consecutive number if existing borings/wells are present. Individual samples will also be designated with a depth code (see below).

Monitoring wells will be numbered consecutively beginning with MW-1.

Test pits will be numbered consecutively beginning with TP-1

Each sample will be given a unique alphanumeric identifier in accordance with the following classification system (such as MW12 0-6 MD, see below):

SAMPLE IDENTIFICATION

LL*	NN*	N-N	LL
Sample Type	Sample Number	Depth Code	QC Identifier

	Solid	Water
Sample Type:	MW - Monitoring Well Boring	
	MW - Monitoring Well	
	SB - Soil Boring	
	SW – Surface Water	
	TP - Test Pit	
	SD – Sediment	
	SS - Surface Soil	
Sample Number:	Number referenced to a sample location map.	
Depth Code:	Depth in feet of sample interval (a=0-0.5, A=0-2, B=2-4, F=10-12, etc.)	
QC Identifier:	FB - Field Blank	
	MS - Matrix Spike	
	TB - Trip Blank	
	MD - Matrix Spike Duplicate	
	WB - Wash or Rinse Blank	
	MB - Matrix Blank	

* L = Letter
* N = Number

Field duplicate samples will be assigned identifiers that do not allow the laboratory to distinguish them as field duplicates. Each sample container will be labeled prior to packing for shipment. The sample identifier, site name, date and time of sampling, and analytical parameters will be written on the label in waterproof ink and recorded in the field book.

16.4.3 SAMPLE CUSTODY

Separate sample custody and documentation procedures will be followed for samples collected for field and laboratory analyses. Components of sample custody are sample labels and chain-of-custody forms.

For laboratory analysis, chain-of-custody forms will be completed for each shipment of samples to track the movement of samples and to provide a written record of all persons handling the samples. The chain-of-custody form will include sample information (sample identification, type, date, and time of collection), analyses requested, and the signature of each person receiving and relinquishing the samples.

The "Remarks" column of the chain-of-custody form will be used to record additional information that may be of use to the laboratory for prescreening the samples. When transferring samples, the individuals relinquishing and receiving the samples will sign, date, and note the time on the chain-of-custody form.

The original chain-of-custody form will accompany the samples to the laboratory. The laboratory will make and maintain a file copy, and the completed original will be returned to the task manager as

a part of the final analytical report. This record serves to document sample custody transfer from the sampler to the shipper, and to the laboratory. Upon receipt of samples, the laboratory will provide a written report to the field investigation manager (or designee) summarizing the condition of samples, sample numbers received and corresponding laboratory numbers, and the estimated date for completion of laboratory analysis.

Sample custody within the laboratory may require an internal chain-of-custody. The sample custody documentation shall include the following:

- Name of associate taking custody of the sample from the sample storage area for preparation or analysis;
- Dates sample removed from and returned to the sample storage area;
- Identification of the tests to be performed on the sample aliquot(s) selected by the associate;
- Sample matrix;
- Laboratory sample numbers; and
- Sample storage location.

Access to the laboratory is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

After the requested analyses on the samples have been completed, any remaining portions of the samples shall be stored for the amount of time required by the project and then disposed of by the laboratory. The disposal of each sample shall be recorded in the laboratory's project file or data management system. Disposal of samples shall occur in accordance with the laboratory procedures after the required retention period.

16.4.4 FIELD QUALITY CONTROL SAMPLES

16.4.4.1 Ambient Blank

The ambient blank consists of ASTM Type II reagent grade water poured into a VOC sample vial at the sampling site. It is handled like an environmental sample and transported to the laboratory for analysis. Ambient blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes.

Ambient blanks are used to assess the potential introduction of contaminants from ambient sources (e.g., active runways, engine test cells, gasoline motors in operation, etc.) to the samples during sample collection. Ambient blanks shall be collected downwind of possible VOC sources.

16.6.4.2 Equipment Blank

An equipment blank is a sample of ASTM Type II reagent grade water poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for

analysis. Equipment blanks are used to assess the effectiveness of equipment decontamination procedures. The frequency of collection for equipment blanks is specified in Table 7-A through Table 7-F. Equipment blanks shall be collected immediately after the equipment has been decontaminated. The blank shall be analyzed for all laboratory analyses requested for the environmental samples collected at the site.

16.6.4.3 Trip Blank

The trip blank consists of a VOC sample vial filled in the laboratory with ASTM Type II reagent grade water, transported to the sampling site, handled like an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes. Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. One trip blank shall accompany each cooler of samples sent to the laboratory for analysis of VOCs.

16.6.4.4 Field Duplicates

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate samples are collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned an identification number in the field such that they cannot be identified (blind duplicate) as duplicate samples by laboratory personnel performing the analysis. Specific locations are designated for collection of field duplicate samples prior to the beginning of sample collection.

Duplicate sample results are used to assess precision of the sample collection process. Precision of soil samples to be analyzed for VOCs is assessed from collocated samples because the compositing process required to obtain uniform samples could result in loss of the compounds of interest. The frequency of collection for field duplicates is specified in Table 7-A through 7-F.

16.6.4.5 Field Replicates

A field replicate sample, also called a split, is a single sample divided into two equal parts for analysis. The sample containers are assigned an identification number in the field such that they cannot be identified as replicate samples by laboratory personnel performing the analysis. Specific locations are designated for collection of field replicate samples prior to the beginning of sample collection. Replicate sample results are used to assess precision. The frequency of collection for field replicates is specified in Table 7-A through 7-F.

16.6.4.6 Environmental Data Reporting: Significant Digits Reflect Quantification Uncertainty

Field measurements of common water-quality parameters, other screening analytical data, calculations of aquifer properties (e.g., hydraulic conductivity, transmissivity, groundwater velocity), and quantities of contaminated soil and water removed and/or treated possess measurable uncertainty or error ranges. When reporting these data, therefore, the number of significant figures employed should reflect the true accuracy and precision (reproducibility) of these measured and calculated values. As a general rule of thumb, field measurements of water quality parameters, quantities of contaminated media removed/remediated, screening analytical data and calculated aquifer properties rarely yield better than two-significant-figure accuracy and precision. Consequently, these field-measured parameters typically should be reported to two significant figures (e.g., DO, 2.1 mg/L; hydraulic conductivity, 120 ft/day; transmissivity, 1,100 ft^2/day; 130 tons of contaminated soil excavated) unless notably low uncertainty exists to justify reporting to three or more significant figures. Manufacturer's performance specifications that document high accuracy and precision for field meters may constitute an example of valid justification for reporting field values to three or more significant figures. Because pH and oxidation-reduction potential are logarithmic values, recommend reporting these parameters to three significant figures. In all cases, standard reporting practice should involve consistency in the number of significant figures used to report measured values.

Definitive analytical data also possess some degree of uncertainty in the final reported values. Only small amounts of definitive data probably possess the required accuracy and precision to be reported to more than three significant figures. The recommendations stated in this section of the MFSP do not set policy for determining the number of significant figures AFCEE laboratories should use in reporting their data. However, contractors and AFCEE staff should constantly recall that the analytical method/analysis is one of the last links in a very long chain of events that forms the foundation of environmental data. Contractors, consequently, are encouraged to use sound scientific judgment in choosing the appropriate number of significant figures and to be consistent in the number of significant figures used to report definitive analytical data in field sampling plans, work plans, site characterization and contamination reports and other IRP documents.

Use of scientifically defensible and consistent numbers of significant figures in reporting analytical and quantitative field data in IRP reports allows the readers and users of these data to properly evaluate measurement uncertainty. This proper evaluation of data accuracy and precision facilitates the scientifically valid interpretation, summarization and subsequent reporting of these data. Contractors are encouraged to comply with *Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications* (ASTM Designation: E 29-02, 2002).

16.5 FIELD MEASUREMENTS

16.5.1 PARAMETERS

The following is a list of all parameters that may be measured during field activities, as well as the equipment that will be used for the measurements:

Parameter	Equipment
Volatiles	MiniRae 2000 (or equivalent)

Water turbidity	Lamotte 2020 Turbidity Meter (or similar)
Water dissolved oxygen	Horiba U22 (or equivalent)
Temperature	Horiba U22 (or equivalent)
Specific Conductivity	Horiba U22 (or equivalent)
Oxygen Reduction Potential (ORP)	Horiba U22 (or equivalent)
pH	Horiba U22 (or equivalent)
Sulfate/sulfide	Hach® DR/850 Portable Colorimeter (or similar)
Nitrite	Hach® DR/850 Portable Colorimeter (or similar)
Alkalinity	Hach® DR/850 Portable Colorimeter (or similar)
Dissolved CO_2	Hach® Digital Titrator (or similar)
Air particulates	Personal Aerosol Monitor

The instruction manuals for the Lamotte 202 Turbidity Meter, the MiniRae 2000, the Horiba U-22, and the Hach instruments are presented in Appendix H. Any additional equipment needed for a specific project or task shall be identified in the SS-WP.

16.5.2 EQUIPMENT CALIBRATION AND QUALITY CONTROL

As required, field analytical equipment shall be calibrated according to the manufacturers' specifications prior to field use. This applies to equipment used for onsite measurements of DO, pH, specific conductance, ORP, and other field parameters. Initial and daily calibrations will be recorded in the field notebook. In addition the reference electrode utilized for ORP and the appropriate conversion factor will be recorded in the field notebook.

16.5.3 EQUIPMENT MAINTENANCE AND DECONTAMINATION

The following decontamination procedure will be followed for equipment to be used for collecting samples for analytical testing:

- Scrub with laboratory-grade detergent (Alconox);
- Rinse with copious quantities distilled water;
- Air dry; and
- Wrap in aluminum foil if being re-used.

Precautions will be taken to minimize any impact to the surrounding area that might result from decontamination operations, and any deviations from these procedures will be documented in the field notebook and on the appropriate sampling record.

Laboratory-supplied sample containers will be cleaned and sealed by the laboratory. The type of container provided and the method of container decontamination will be documented in the laboratory's permanent record of the sampling event.

16.5.4 FIELD MONITORING MEASUREMENTS

16.5.4.1 Groundwater Level Measurements

Water-level measurements shall be taken in all wells and piezometers to determine the elevation of the water table or piezometric surface at least once within a single 24-hour period. These measurements shall be taken after all wells and piezometers have been installed and developed and their water levels have recovered completely. Any conditions (e.g., barometric pressure) that may affect water levels shall be recorded in the field log. The field log shall also include the previous water level measurement for each well (to determine if current water level is reasonable).

Water-level measurements shall be taken with electric sounders, air lines, pressure transducers, or water-level recorders (e.g., Stevens recorder). Devices that may alter sample composition shall not be used. Pressure gauges, manometers, or equivalent devices shall be used for flowing wells to measure the elevation of the piezometric surface. All measuring equipment shall be decontaminated according to the specifications in Section 7.3 and 5.12. Ground-water level shall be measured to the nearest 0.01 foot. (Two or more sequential measurements shall be taken at each location until two measurements agree to within + or - 0.01 foot.)

Static water levels shall be measured each time a well is sampled, and before any equipment enters the well. If the casing cap is airtight, allow time prior to measurement for equilibration of pressures after the cap is removed. Repeat measurements until water level is stabilized.

16.5.4.2 Floating Hydrocarbon Measurements

The thickness of hydrocarbons floating in monitor wells shall be measured with an electronic interface probe. Hydrocarbon detection paste, or any other method that may affect water chemistry, shall not be used. When detected, the presence of floating hydrocarbons shall be confirmed by withdrawing a sample with a clear, bottom-fill Teflon® bailer.

16.5.4.3 Groundwater Discharge Measurements

Groundwater discharge measurements shall be obtained during monitor well purging and aquifer testing. Groundwater discharges may be measured with orifice meters, containers of known volume, in-line meters, flumes, or Weirs, following the guidelines specified in the *Water Measurement Manual*, Bureau of Reclamation, 1967. Measurement devices shall be calibrated using containers of known volume.

16.5.4.4 Sulfate/Sulfide Measurements

Sulfide concentrations in groundwater cannot be measured using a probe and will be analyzed in the field via colorimetric analysis with a Hach® DR/850 Portable Colorimeter (or similar) after appropriate sample preparation. USEPA-approved Hach® Method 8131 (0 to 0.70 mg/L) will be used to analyze for sulfide. The manual for the colorimeter, including calibration procedures, and the procedures for Method 8131 are contained in Appendix H.

16.5.4.5 Nitrite Measurements

Nitrite concentrations in groundwater cannot be measured using a probe, so a Hach® DR/850 Portable Colorimeter (or similar) and Hach Method 8507 (0 to 0.350 mg/L) will be utilized in the field to

determine these concentrations. The manual for the colorimeter, including calibration procedures, and the procedures for Method 8507 are contained in Appendix H.

16.5.4.6 Alkalinity Measurements

Alkalinity of the groundwater sample will be measured in the field by via titrimetric analysis using USEPA-approved Hach® Method 8221 (0 to 5,000 mg/L as calcium carbonate), or equivalent. The procedures for this method are contained in Appendix H.

16.5.4.7 Portable Photoionization Analyzer

The photoionization analyzer will be a RaeSystems MiniRae 2000 (or equivalent), equipped with a 10.6 eV lamp. The MiniRae is capable of ionizing and detecting compounds with an ionization potential of less than 10.6 eV. This accounts for up to 73% of the volatile organic compounds on the Target Compound List.

Calibration must be performed at the beginning and end of each day of use with a standard calibration gas having an approximate concentration of 100 parts per million of isobutylene. If the unit experiences abnormal perturbation or erratic readings, additional calibration will be required.

All calibration data must be recorded in field notebooks and on calibration log sheets to be maintained on-site.

A battery check must be completed at the beginning and end of each working day.

16.5.4.8 Personal Aerosol Monitor

The operator shall ensure that the instruments respond properly to the substances that they are designed to monitor. Real time aerosol monitors must be zeroed at the beginning of each sampling period. The specific instructions for calibration and maintenance provided for each instrument should be followed.

All calibration data must be recorded in field notebooks and on calibration log sheets to be maintained on-site.

A battery check must be completed at the beginning and end of each working day.

16.5.4.9 PH Meter

Calibration of the pH meter must be performed at the start of each day of use, and after very high or low readings as required by this plan, according to manufacturer's instructions.

National Institute of Standards and Technology - traceable standard buffer solutions which bracket the expected pH range will be used. The standards will be pH of 4.0, 7.0 and 10.0 standard units.

The use of the pH calibration must be used to set the meter to display the value of the standard being checked. The calibration data must be recorded on calibration sheets maintained on-site or with the piece of equipment.

16.5.4.10 Specific Conductivity Meter and Temperature Probe

Calibration checks using the conductivity standard must be performed at the start of each day of use, after five to ten readings or after very high or low readings as required by this plan, according to manufacturer's instructions.

The portable conductivity meter must be calibrated using a reference solution of 200 uohms/cm on a daily basis. Readings must be within five percent to be acceptable. The thermometer of the meter must be calibrated against the field thermometer on a weekly basis.

16.5.4.11 Turbidity, Dissolved Oxygen, Dissolved CO_2, and Oxygen Reduction Potential Meters

These meters must be checked at the start of each day of use and at the end of the day according to manufacturer's instructions.

16.5.5 FIELD PERFORMANCE AND SYSTEM AUDITS

Field activities will be monitored on a per-task basis by the Technical Director or his/her designee to ensure compliance with this SAP and the SS-WP. Each task or project will be monitored at least once in the field.

16.6 RECORD KEEPING

Bound field logbooks will be maintained by the field supervisor and other team members to provide a daily record of significant events, observations, and measurements during the field investigation. All entries will be signed and dated. All information pertinent to the field survey and/or sampling will be recorded in the logbooks. The logbooks will be bound, with sequentially numbered pages. Waterproof ink will be used in making all entries. Entries in the logbook will include, at a minimum, the items listed below:

General information:

- Names and titles of author and assistants;
- Date and time of entry;
- Physical/environmental conditions during field activity;
- Purpose of sampling activity;
- Location of sampling activity; and
- Names and titles of field crew.

Sampling documentation:

- Sample medium (e.g., groundwater, soil);
- Description of sampling point(s);

- Date and time of collection;
- Sample identification; and
- Sample analyses and containers.

Other information:

- Names and titles of site visitors;
- Field observations (i.e., unusual field conditions);
- Field measurements (such as pH, conductivity, temperature) and specific instrument calibration data;
- Field equipment (make, model, serial number);
- Equipment decontamination frequency; and
- Sample handling (e.g., preservation with ice) and shipping (i.e., shipping company, air bill number) information.

None of the field logbooks or forms will be destroyed or discarded, even if they are illegible or contain inaccuracies that require a replacement document. If a previously recorded entry is discovered to be incorrect, the incorrect entry will be crossed out in such a manner that it is still legible. The correct entry will be written in, and the change will be initialed and dated. If the change is made by someone other than the original author, or if the change is made on a subsequent day, a reason for the change will be recorded at the current active location in the logbook, with cross references.

The contractor shall maintain field records sufficient to recreate all sampling and measurement activities and to meet all ERPIMS data loading requirements. The requirements listed in this section apply to all measuring and sampling activities. Requirements specific to individual activities are listed in the section that addresses each activity. The information shall be recorded with indelible ink in a permanently bound notebook with sequentially numbered pages. These records shall be archived in an easily accessible form and made available to the Air Force upon request.

17 REFERENCES

Air Force Center for Environmental Excellence (AFCEE). 2005. Guidance for Contract Deliverables, Appendix C: Quality Assurance Project Plan (QAPP), Version 4.0.

Air Force Center for Environmental Excellence (AFCEE). 2002. Guidance for Contract Deliverables, Appendix B: Model Field Sampling Plan, Version 1.2.

Army, United States Environmental Protection Agency (USEPA) Region 2, New York State Department of Environmental Conservation (NYSDEC). 1993. Federal Facilities Agreement (FFA).

American Society for Testing and Materials. 1993. ASTM *Annual Book of Standards*.

American Society for Testing and Materials (ASTM). 2002. *Standard Practices for Using Significant Digits in Test Data to Determine Conformance with Specifications.*

New York State Department of Environmental Conservation (NYSDEC). 2002. Draft DER-10 Technical Guidance for Site Investigation and Remediation.

New York State Department of Environmental Conservation (NYSDEC). 2001. Technical Guidance for Site Investigation and Remediation SW-96-09: Development and Review of Site Analytical Plans.

New York State Department of Environmental Conservation (NYSDEC). 2000. Analytical Service Protocols.

New York State Department of Environmental Conservation (NYSDEC). 1998 with 2000 and 2004 Addendum. Ambient Water Quality Standard and Guidance Values and Groundwater Effluent Limitations.

New York State Department of Environmental Conservation (NYSDEC). 1994. Technical and Administrative Guidance Memorandum (TAGM) HWR-94-4046.

Parsons. 1995. Final Generic Installation Remedial Investigation/Feasibility Study (RI/FS) Workplan.

United States Army Corps of Engineers (USACE). 2001. Requirements for the Preparation of Sampling and Analysis Plan, USACE EM200-1-3.

United States Army Corps of Engineers (USACE). 1997. Chemical Quality Assurance for Hazardous, Toxic and Radioactive Waste (HTRW) Project, USACE EM200-1-6.

United States Environmental Protection Agency (USEPA). National Drinking Water Regulations. On-line resources available at http://www.epa.gov/safewater/mcl.html.

United States Environmental Protection Agency (USEPA). Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846) – Third Edition, September 1986; Final Update I, July 1992; Final Update IIA, August 1993; Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

United States Environmental Protection Agency (USEPA). 2004. Uniform Federal Policy for Quality Assurance Project Plans, Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs.

United States Environmental Protection Agency (USEPA). 2004. USEPA National Functional Guidelines for Inorganic Data Review.

United States Environmental Protection Agency (USEPA). 2004. ProUCL Version 3.0 User Guide. April.

United States Environmental Protection Agency (USEPA). 2003. Recommendations of the Technical Review Workgroup for Lead for an Interim Approach to Assessing Risks Associated with Adult Exposures to Lead in Soil.

United States Environmental Protection Agency (USEPA). 2002. National Recommended Water Quality Criteria: November.

United States Environmental Protection Agency (USEPA). 2002. Guidance for Quality Assurance Project Plans. QA/G-5.

United States Environmental Protection Agency (USEPA). 2002. Guidance on Choosing a Sampling Design for Environmental Data Collection. QA/G-5S.

United States Environmental Protection Agency (USEPA). 2001. EPA Requirements for Quality Assurance Project Plans. QA/R-5.

United States Environmental Protection Agency (USEPA). 2001. Quality Management Plan for Western Ecology Division.

United States Environmental Protection Agency (USEPA). 2001. Guidance for Preparing Standard Operating Procedures. QA/G-6.

United States Environmental Protection Agency (USEPA). 2000. Guidance for Data Quality Assessment: Practical Methods for Data Analysis. 9QA/G-9.

United States Environmental Protection Agency (USEPA). 2000. Guidance for the Data Quality Objectives Process. QA/G-4.

United States Environmental Protection Agency (USEPA). 2000. Data Quality Objectives Process for Hazardous Waste Site Investigations. QA/G-4HW.

United States Environmental Protection Agency (USEPA). 1999. USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review.

United States Environmental Protection Agency (USEPA). 1995. 2185 - Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations with Implementation Guidance.

United States Environmental Protection Agency (USEPA). 1994. Guidance Manual for the Integrated Exposure Uptake Biokinetic Model for Lead in Children. February.

United States Environmental Protection Agency (USEPA). 1991. Preparation Aids for the Development of Category I Quality Assurance Project Plans. EPA/600/8-91/003.

United States Environmental Protection Agency (USEPA). 1979. *Methods for Chemical Analysis of Water and Waste*.

United States Environmental Protection Agency (USEPA) Region 2. 2004. Guidance for the Development of Quality Assurance Project Plans for Environmental Monitoring Projects.

United States Environmental Protection Agency (USEPA) Region 2. *Region 2 RCRA and CERCLA Data Validation Standard Operating Procedures (SOPs). On-line resources at http://www.epa.gov/region02/desa/hsw/sops.htm*.

United States Environmental Protection Agency (USEPA) Region III. 2004. Risk-Based Concentration (RBC) Table. On-line database last updated October. http://www.epa.gov/reg3hwmd/risk/index.htm

United States Environmental Protection Agency (USEPA) Region 6. Region 6 Human Health Medium-Specific Screening Levels 2004-2005. On-line resource last updated December 2004 http://www.epa.gov/Arkansas/6pd/rcra_c/pd-n/screen.htm.

United States Environmental Protection Agency (USEPA) Region 9. 2004. Preliminary Remediaiton Goals. On-line resources available at http://www.epa.gov/region09/waste/sfund/prg/index.htm, last updated December.

Appendix A

Applicable SAP Guidance Cross Reference Table

Appendix A
Applicable SAP Guidance Cross Reference Table
Sample Analysis Plan
Seneca Army Depot Activity
Romulus, New York

Section No	Sub-section No.	Title	AFCEE	NYSDEC		USEPA, USEPA Region 2				USACE	
			AFCEE, Guidance for Contract Deliverables, Appendix C	NYSDEC DER-10	NYSDEC, TAGM SW-96-09	QMP for WED, Appendix 3	EPA QA/R-5	EPA QA/G-5 Region 2 Guidance, 2004	UFP-QAPP, 2004	EM200-1-3	EM200-1-6
		Title and cover					3.2.1 (A1)	A1	2.1	3.3.5.1	
		ACRONYMS								3.3.6	
		Cover Letter					3.2.3 (A3)	A3			
1		Introduction	1						2.2.4		
2	2.1	Seneca Program Background	2.1			Project Management			2.2.4		
	2.2	Project Background	2.2			Project Management			2.2.4		
	2.3	Project Scope and Objectives	2.3	2.2-1		Project Management			2.5.1		
	2.4	Applicable Regulations									
3		Program Organization and Responsibilities	3	2.1(a), QAO 2.2-2		Project Management	3.2.4 (A4)	A4	1.3 2.4.1 2.4.2 2.4.3	3.3.5.3 3.3.5.4	
4	4.1	DQO			III-B-1	DQOs	3.2.7 (A7)	A7	2.2.4 2.6.1		
	4.2	Data Types	4.1								
	4.3	Data Quality Indicators	4.2		III-B-1	DQOs	3.3.5 (B5)	A7	2.6.2	3.3.5.5.2 3.3.5.8	
	4.4	Reporting limits	4.3				3.3.7 (B7)		2.6.2 3.2.2	3.3.5.7.2	
	4.5	Quality Control Activities	4.4	2.1(b)	III-B-2		3.3.5 (B5)	B5	2.6.2 3.4	3.3.5.7.3	
	4.6	Quality Control Checks	4.5				3.3.8 (B8)	B8	2.6.2 3.1.2.5 3.2.4		
5	5.1	Field Sampling Procedure	5.1								
	5.2	Sample Collection Documentation							3.3.1		
	5.3	Sample Handling and Custody	5.2	2.2-6		Measurement/Data Acquisition	3.3.3 (B3)		3.3.2 3.3.3	3.3.5.6	
6	6	Screening Analytical Methods	6	2.1(c), 2.1(g)		Measurement/Data Acquisition		B4	3.2.1	3.3.5.7	

Appendix A
Applicable SAP Guidance Cross Reference Table
Sample Analysis Plan
Seneca Army Depot Activity
Romulus, New York

Section No	Sub-section No.	Title	AFCEE	NYSDEC		USEPA, USEPA Region 2				USACE	
			AFCEE, Guidance for Contract Deliverables, Appendix C	NYSDEC DER-10	NYSDEC, TAGM SW-96-09	QMP for WED, Appendix 3	EPA QA/R-5	EPA QA/G-5 Region 2 Guidance, 2004	UFP-QAPP, 2004	EM200-1-3	EM200-1-6
7	7	Definitive Data Analytical Methods	7	2.1(c), 2.1(e), 2.1(h),(i) 2.1(j) 2.1 (k) (l) 2.1(m)		Measurement/Data Acquisition		B4	3.2.1	3.3.5.7	
8		Data Management	8			Data Management	3.3.10 (B10)				
	8.1	Data Review Screening Data	8.1						3.5.2.1 3.5.4 5.1 5.2.1		
	8.2	Data Review Definitive Data	8.2	2.1(d), 2.1(f)			3.2.9 (A9)	D1	3.5.2.3 3.5.3 3.5.4 5.1 5.2.1	3.3.5.9 3.3.5.10.2	2-1 2-3 2-4 3-1 3-3
	8.3	Data reduction				Data Management		B3	3.5.4	3.3.5.8	
	8.4	Data assessment procedure							5.1	3.3.5.10.1	
	8.5	Data Verification	8.2 8.6			Data Management					
	8.6	Data Validation	8.6			Data Validation and usability	3.5.1 (D1) 3.5.2 (D2) 3.5.3 (D3)	D2	3.5.4 5.1 5.2.2	3.3.5.10.2 3.3.5.10.4	3-1 3-3
	8.7	Data Usability Assessment	8.6						5.2.3		
	8.8	Non-direct Measurement Data Evaluation						B9	2.7		
	8.9	Reconciliation						D3		3.3.5.10.3	
	8.10	Electronic data reports	8.4	2.2-7			3.2.9 (A9)		3.5.3	3.3.5.9	2-2
	8.11	Archiving	8.5 8.6 8.7			Data Management			3.5.4 3.5.5	3.3.5.9	
	8.12	Hardcopy data reporting format	8.8						3.5.3	3.3.5.9	2-2

Appendix A
Applicable SAP Guidance Cross Reference Table
Sample Analysis Plan
Seneca Army Depot Activity
Romulus, New York

Section No	Sub-section No.	Title	AFCEE	NYSDEC		USEPA, USEPA Region 2				USACE	
			AFCEE, Guidance for Contract Deliverables, Appendix C	NYSDEC DER-10	NYSDEC, TAGM SW-96-09	QMP for WED, Appendix 3	EPA QA/R-5	EPA QA/G-5 Region 2 Guidance, 2004	UFP-QAPP, 2004	EM200-1-3	EM200-1-6
	8.13	Data Analysis							3.5.4		
9		Performance and Audits	9			Assessment/Oversight	3.3.6 (B6) 3.4.1 (C1)	C1	4.1.1 4.1.2	3.3.5.7.4	
10		Preventive Maintenance	10				3.3.6 (B6)	B7	3.1.2.4 3.2.3	3.3.5.7.1	
11		Nonconformance /Corrective Actions	11						1.2.7 4.1.2	3.3.5.7.5	
12		QA Reports to Management	8.3 12			QA Reports	3.2.9 (A9)	C2	4.2		4
13		Revisions and Distribution				Updates and Revision	2.7 3.2.9 (A9) 3.2.3 (A3)	A3	1.2.7 1.2.8 2.2.2 2.3.1 2.3.2	3.3.5.2	
14		Special Training/Certification					3.2.8 (A8)	A8	2.4.4		
15		Documents and Records					3.2.9 (A9)	A9	1.2.8 3.5.1		
16		Field Sampling Plan		2.2-3, 2.2-6,	III-B-3	Measurement/Data Acquisition	3.3.2 (B2)		3.1 3.3.2.2 Appendix A, SOP	3.3.4	
17		References	13								
Appendix A		Cross Reference table						A2			
Appendix C		Sign-Off Sheet							2.3.2		
Appendix D, Figure 3		Organization Chart					A4	A4	1.3 2.4.1	3.3.5.3 3.3.5.4	
Appendix E		Tables					3.3.2 (B2) 3.3.5 (B5)	B2 B3	3.1.2.2 3.4	3.3.5.7	
site-specific work plan				2.2-4, 2.2-5,	III-B-2	Measurement/Data Acquisition	3.2.5 (A5) 3.2.6 (A6) 3.3.4 (B4) 3.3.5 (B5)	A7 A8 B1 B2 B9 D3	2.5 2.6 2.8.1 2.8.2	3.3.5.5.1	

References:

1. AFCEE. 2005. Guidance for Contract Deliverables, Appendix C: Quality Assurance Project Plan (QAPP), Final Version 4.0
2. NYSDEC. 2002. Draft DER-10. Technical Guidance for Site Investigation and Remediation
3. NYSDEC. 2001. Technical Administrative Guidance Memorandum SW-96-09. Development and Review of Site Analytical Plans.

Appendix A
Applicable SAP Guidance Cross Reference Table
Sample Analysis Plan
Seneca Army Depot Activity
Romulus, New York

Section No	Sub-section No.	Title	AFCEE	NYSDEC		USEPA, USEPA Region 2				USACE	
			AFCEE, Guidance for Contract Deliverables, Appendix C	NYSDEC DER-10	NYSDEC, TAGM SW-96-09	QMP for WED, Appendix 3	EPA QA/R-5	EPA QA/G-5 Region 2 Guidance, 2004	UFP-QAPP, 2004	EM200-1-3	EM200-1-6

4. USEPA. 2001. Quality Management Plan for Western Ecology Division.
5. USEPA. 2001. EPA Requirements for Quality Assurance Project Plans. EPA QA/R-5.
6. USEPA. 2002. Guidance for Quality Assurance Project Plans. EPA QA/G-5.
7. USEPA Region 2. 2004. Guidance for the Development of Quality Assurance Project Plans for Environmental Monitoring Projects.
8. USEPA. 2004. Uniform Federal Policy for Quality Assurance Project Plans.
9. USACE. 2001. Requirements for the Preparation of Sampling and Analysis Plans. EM200-1-3.
10. USACE. 1997. Chemical Quality Assurance for Hazardous, Toxic and Radioactive Waste (HTRW) Projects. EM200-1-6.

Appendix B

Standard Forms

Figure 7-4

QUALITY CONTROL FIELD AUDIT REPORT

SUMMARY INFORMATION

1. PROJECT NAME: _____

2. PROJECT ADDRESS: _____

_____

_____

3. PRELIMINARY ASSESSMENT _____ RI/FS _____ RD _____ CONSTRUCTION_____

OTHER _____

4. DATE(S) OF QC FIELD AUDIT _____

5. AUDITOR'S NAME _____PHONE_____

6. FACILITY CONTACT _____PHONE_____

7. CONTRACTOR CONTACT_____PHONE_____

8. PERSONNEL ON-SITE

NAME	REPRESENTING	PHONE

9. AUDITOR'S COMMENTS

_____

_____

_____

_____

10. WEATHER CONDITIONS

SUNNY ; PARTLY SUNNY ; PARTLY CLOUDY ; CLOUDY ; RAIN ; DRIZZLE ; SNOW ; SLEET

TEMPERATURE_____ WIND SPEED_____ WIND DIRECTION_____

11. LEVEL OF PERSONNEL PROTECTION REQUIRED IN WORK PLAN

A B C D

LEVEL OF PERSONNEL PROTECTION ACTUALLY DONNED:

A B C D

12. FIELD SURVEY EQUIPMENT

INSTRUMENT	MODEL	CALIBRATION CHECK	CALIBRATION STANDARD	SPAN SETTING
CONDUCTIVITY METER				
DISSOLVED O_2 METER				
PH METER				
COMBUSTIBLE GAS INDICATOR (LEL/O_2)				
FLAME IONIZATION DETECTOR (OVA)				
PHOTOIONIZATION DETECTOR (HNU)				
TOTAL GAS INDICATOR (CO,H_2S)				
OTHER				

OBSERVATIONS _____

_____

_____

13. DID THE SAMPLING TEAM TAKE PERIODIC SURVEYS OF THE AMBIENT AIR CONDITIONS?

YES NO N/A

14. DID THE SAMPLING TEAM PROVIDE A DECON ZONE DESIGNATING CLEAN AND CONTAMINATED AREAS?

YES NO N/A

15. WERE PHOTOGRAPHS TAKEN? YES NO

16. AUDITOR'S COMMENTS

_____

_____

_____

MONITORING WELL SAMPLING SETUP AND EVACUATION

EVACUATION PROCEDURES

1. WELL CASING CONSTRUCTION STAINLESS STEEL TEFLON PVC OTHER _____

2. DIAMETER OF WELL CASING 2" 4" 6" OTHER _____

3. LOCKING CAPS ON THE WELLS? YES NO N/A PROTECTIVE CASING? YES NO N/A

4. METHOD UTILIZED TO DETERMINE THE STATIC WATER LEVEL

WATER LEVEL INDICATOR OTHER _____

5. REFERENCE POINT THAT THE STATIC WATER LEVEL WAS MEASURED FROM:

SURVEY POINT	TOP OF INNER CASING	TOP OF PROTECTIVE CASING	HEIGHT OF CASING ABOVE GROUND SURFACE

6. WAS THE WATER LEVEL INDICATOR DECONTAMINATED ACCORDING TO STANDARD PROCEDURES BETWEEN EACH WELL?
YES NO N/A

IF NO, METHOD USED: _____

_____

_____

7. EVACUATION METHOD:

BAILER CENTRIFUGAL PUMP PERISTALTIC PUMP BLADDER PUMP SUBMERSIBLE PUMP

GAS DISPLACEMENT PUMP GAS LIFT PUMP OTHER _____

8. TYPE OF HOSE UTILIZED:

POLYETHYLENE TEFLON SILASTIC N/A OTHER _____

9. WAS THE HOSE DEDICATED TO EACH WELL LOCATION? YES NO N/A

IF NO, METHOD OF DECONTAMINATION _____

10. WAS THE PUMP DEDICATED TO EACH WELL LOCATION? YES NO N/A

11. WAS THE PUMP: LABORATORY DECONTAMINATED? FIELD DECONTAMINATED? N/A

12. WAS THE PUMP DECONTAMINATED ACCORDING TO STANDARD PROCEDURES?

YES NO IF NO, METHOD OF DECONTAMINATION _____

13. WAS THE PUMP HEAD OR END OF HOSE WITHIN 6 FEET OF THE DYNAMIC WATER LEVEL DURING EVACUATION?
YES NO N/A

14. WAS THE DECONTAMINATION AREA LOCATED AWAY FROM THE SOURCE OF CONTAMINATION?

YES NO N/A

15. AUDITOR'S COMMENTS

_____

_____

_____

AQUEOUS SAMPLING PROCEDURES

1. AQUEOUS MATRIX SAMPLED:

POTABLE WELL GROUND WATER SURFACE WATER LEACHATE RUNOFF STORM SEWER

SANITARY SEWER OTHER _____

2. TYPE OF SAMPLE: GRAB COMPOSITE IF COMPOSITE - SAMPLES/COMPOSITE _____

3. WAS THE VOA SAMPLE COLLECTED FIRST? YES NO N/A

4. TYPE OF SAMPLING EQUIPMENT:

	MATERIAL OF CONSTRUCTION			
	STAINLESS STEEL	TEFLON	GLASS	OTHER
BAILER	_____	_____	_____	_____
BLADDER PUMP	_____	_____	_____	_____
SAMPLER	_____	_____	_____	_____
COLIWASA	_____	_____	_____	_____
KEMMERER DEPTH SAMPLER	_____	_____	_____	_____
WHEATON DIP SAMPLER	_____	_____	_____	_____
TUB SAMPLER	_____	_____	_____	_____
BACON BOMB	_____	_____	_____	_____

5. TYPE OF LEADER LINE THAT COMES IN CONTACT WITH THE WELL WATER:

TEFLON TEFLON COATED STAINLESS STEEL N/A OTHER _____

6. LENGTH OF THE LEADER LINE _____

7. WAS THE SAMPLING EQUIPMENT DEDICATED? YES _____ NO _____

8. WAS THE SAMPLING EQUIPMENT: LAB DECONTAMINATED? FIELD DECONTAMINATED?

9. WAS THE SAMPLING EQUIPMENT DECONTAMINATED ACCORDING TO STANDARD PROCEDURES?

YES NO IF NO, METHOD OF DECONTAMINATION: _____

10. WAS THE DECONTAMINATION AREA LOCATED AWAY FROM THE SOURCE OF CONTAMINATION?

YES NO

11. ARE DISPOSABLE GLOVES WORN AND CHANGED BETWEEN EACH SAMPLE LOCATION? YES NO

12. AUDITOR'S COMMENTS:

_____

_____

_____

FIGURE 7-5

NONCONFORMANCE AND CORRECTIVE ACTION REPORT

Date _____

NCR No. _____

Description of Nonconformance and Cause _____
_____
_____
_____
_____
_____

Proposed Disposition _____
_____
_____
_____
_____
_____

Submitted by: _____ Date: _____
Approved by: _____

DISPOSITION (by Project Manager or Designee)

Implementation of Disposition Assigned to: _____

Actual Disposition _____
_____
_____
_____
_____
_____

Disposition completed on: _____
Date

_____
Signature

VERIFICATION
Disposition reviewed and work inspected by: _____ on _____
Disposition verified by: _____ on _____
(Use additional sheet or memo if necessary)

FIGURE 10-1

DATE _____

MMRP: (Installation name)
DAILY QUALITY CONTROL REPORT

DAY	S	M	T	W	TH	F	S

USACE PROJECT MGR. _____

PROJECT _____

JOB NO. _____

CONTRACT NO. _____

WEATHER	BRIGHT SUN	CLEAR	OVERCAST	RAIN	SNOW
TEMPERATURE	< 32	32 - 50	50 - 70	70-85	>85
WIND	STILL	MODERATE	HIGH	REPORT NO.	
HUMIDITY	DRY	MODERATE	HUMID		

SUBCONTRACTORS ON-SITE:
EQUIPMENT ON SITE:
WORK PERFORMED (INCLUDING SAMPLING):
QUALITY CONTROL ACTIVITIES (INCLUDING FIELD CALIBRATIONS):
HEALTH AND SAFETY LEVELS AND ACTIVITIES:
PROBLEMS ENCOUNTERED/CORRECTIVE ACTION TAKEN:
SPECIAL NOTES:
TOMORROW'S EXPECTATIONS:

BY _____ TITLE _____

QA/QC INFORMATION

1. LABORATORY:

NAME _____ PHONE _____

CONTACT PERSON _____

CLP CLP CAPABLE CERTIFIED OTHER _____

3. SAMPLE INFORMATION:

MATRIX	PARAMETER	PRESERVATIVE	CONTAINER DESCRIPTION

3. WHAT ORDER BY ANALYTICAL PARAMETER ARE SAMPLES COLLECTED: _____

_____

4. FIELD BLANKS: YES NO N/A FREQUENCY _____

METHOD: _____

WAS IDENTICAL BOTTLE TO BOTTLE TRANSFER OF WATER UTILIZED? YES NO

5. TRIP BLANKS: YES NO N/A FREQUENCY _____

6. WHAT WAS THE SOURCE OF THE BLANK WATER? LABORATORY DEMONSTRATED ANALYTE-FREE
OTHER_____

7. SAMPLE PACKAGING AND HANDLING:

SAMPLE CONTAINERS LABELED	YES	NO	N/A
COC FORMS COMPLETED	YES	NO	N/A
CUSTODY SEALS	YES	NO	N/A
SAMPLES PRESERVED TO 4°C:	YES	NO	N/A

8. AUDITOR'S COMMENTS

_____

_____

_____

_____

AFCEE
SCREENING DATA SHEET 1
DATA PACKAGE

Analytical Method: _____ **Contract #:** _____

Base/Command: _____ **Prime Contractor:** _____

Field Sample ID

_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Comments:

_____
_____
_____

Signature: _____ Name: _____

Date: _____ Title: _____

AFCEE FORM S-1

AFCEE
SCREENING DATA SHEET 2
RESULTS

Analytical Method: _____

Contract #: _____ **Field Sample ID:** _____

Matrix: _____ **Date Analyzed:** _____

Concentration Units (µg/L, mg/kg dry weight or °C): _____

Analyte/Test	MDL	RL	Result	Qualifier

Comments: _____

AFCEE FORM S-2 Page _____ of _____

AFCEE
SCREENING DATA SHEET 3
FIELD DUPLICATES

Analytical Method: _____ **Contract #:** _____

Units: _____

Analyte/Test	Sample Result	Duplicate Sample Result	%D or %RPD	Acceptance Criteria	Q

Comments: _____

AFCEE FORM S-3 Page _____ of _____

Appendix C

Project Personnel Sign-Off Sheet

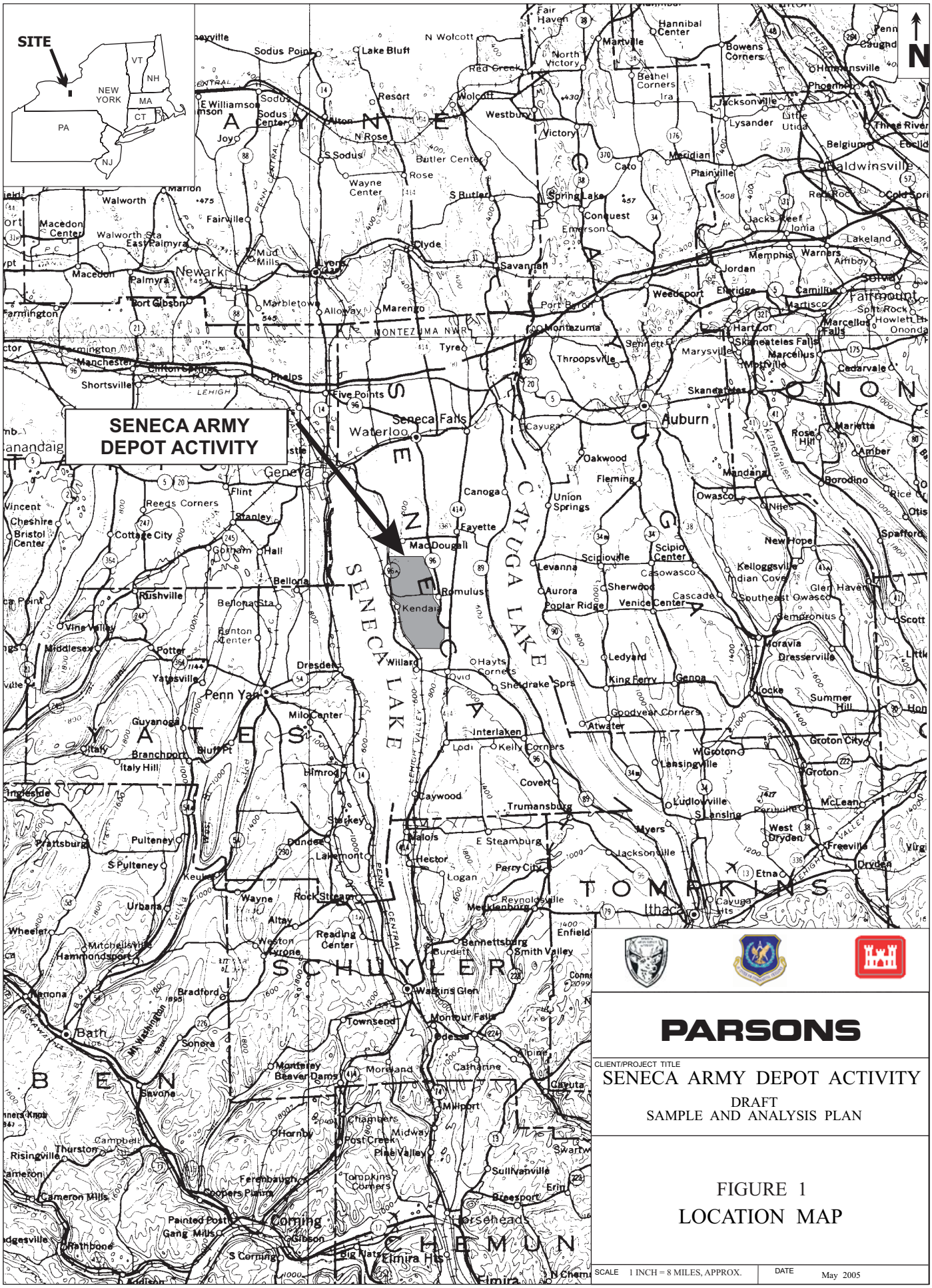
Appendix C
Project Personnel Sign-Off Sheet

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Todd Heino	Project Manager	(617)-449-1405		
Jeff Adams	Task Order Manager	(617)-449-1570		
Jackie Travers	Task Order Manager	(617)-449-1566		
James Lowerre	Quality Assurance Officer	(617)-449-1559		
Tom Andrews	Field Team Leader	(716) 633-7074		
Katherine Lapierre	Project Chemist	(512) 719-6000x6806		
David Miller	STL Pittsburgh, Project Manger	(412)963-7058		
Tony Bogolin	STL Buffalo, Project Manager	(716) 691-2600		
Nancy Mattern	GEL, Charlestown, SC, Project Manger	(843) 556-8171		
Mark Wilson	CAS, Rochester, NY, Project Manger	(585)288-5380		
TBD	Subcontractor project manager			

All the above identified personnel or any other key project personnel should read the appropriate sections of the approved SAP and perform the tasks as described. The signed sheets should be forwarded to Kaaren Godin at 150 Federal Street, Boston, MA 02110 (email: kaaren.godin@parsons.com) for the central project file.

Appendix D

Figures



PARSONS

CLIENT/PROJECT TITLE

SENECA ARMY DEPOT ACTIVITY

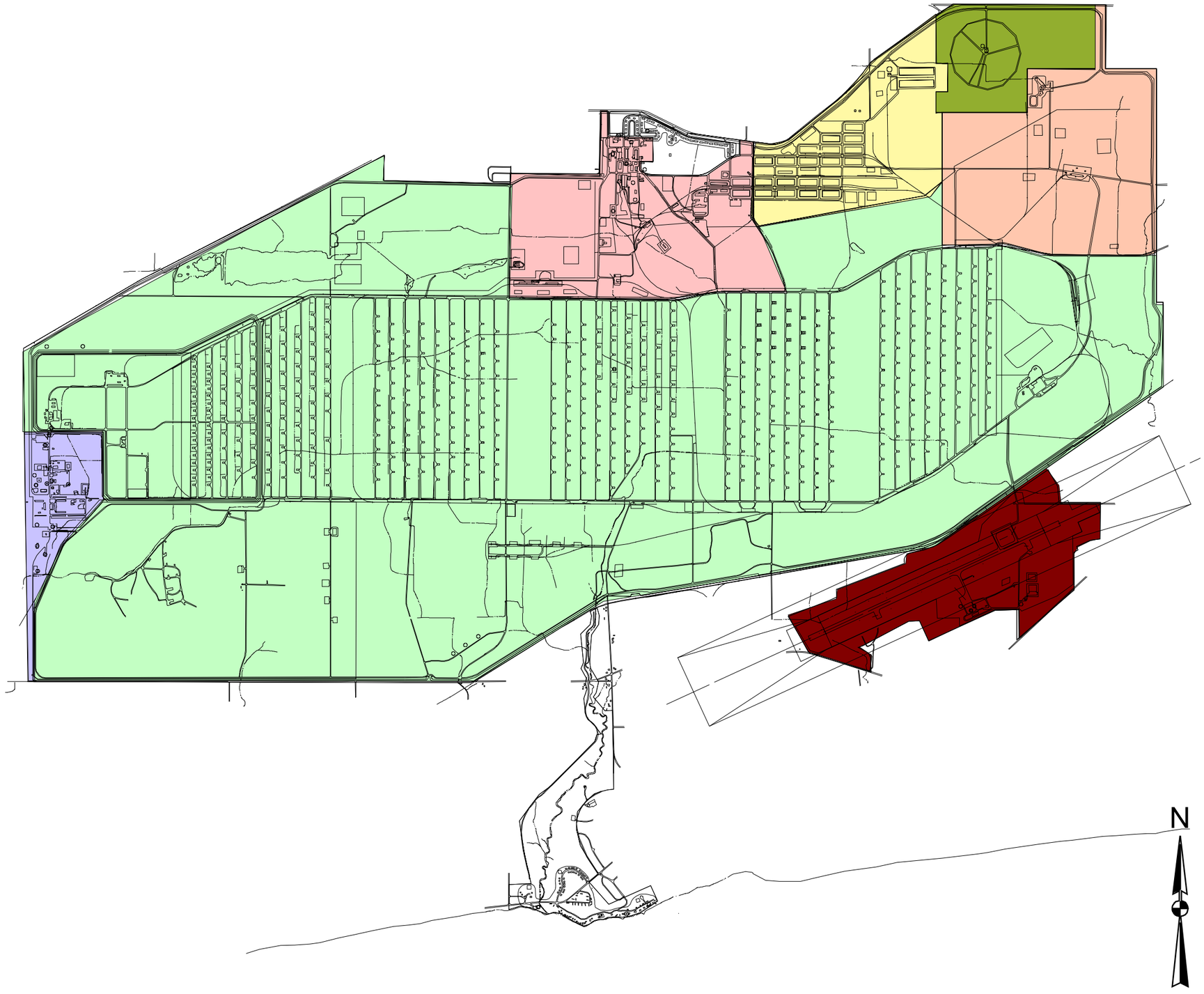
DRAFT
SAMPLE AND ANALYSIS PLAN

FIGURE 1
LOCATION MAP

SCALE 1 INCH = 8 MILES, APPROX.

DATE May 2005

P:\PIT\PROJECTS\SENECA\HUNTSVILLE HTW\\LOCMAP1.CDR



LEGEND

Intended Land Use

- Airfield
- Conservation/Recreation
- Fed to Fed Transfer
- Industrial
- Institutional
- Prison
- Warehouse

0.2 0 0.2 0.4 0.6 0.8 Miles

PARSONS

SENECA ARMY DEPOT ACTIVITY
ROMULUS, NEW YORK
SAMPLE AND ANALYSIS PLAN

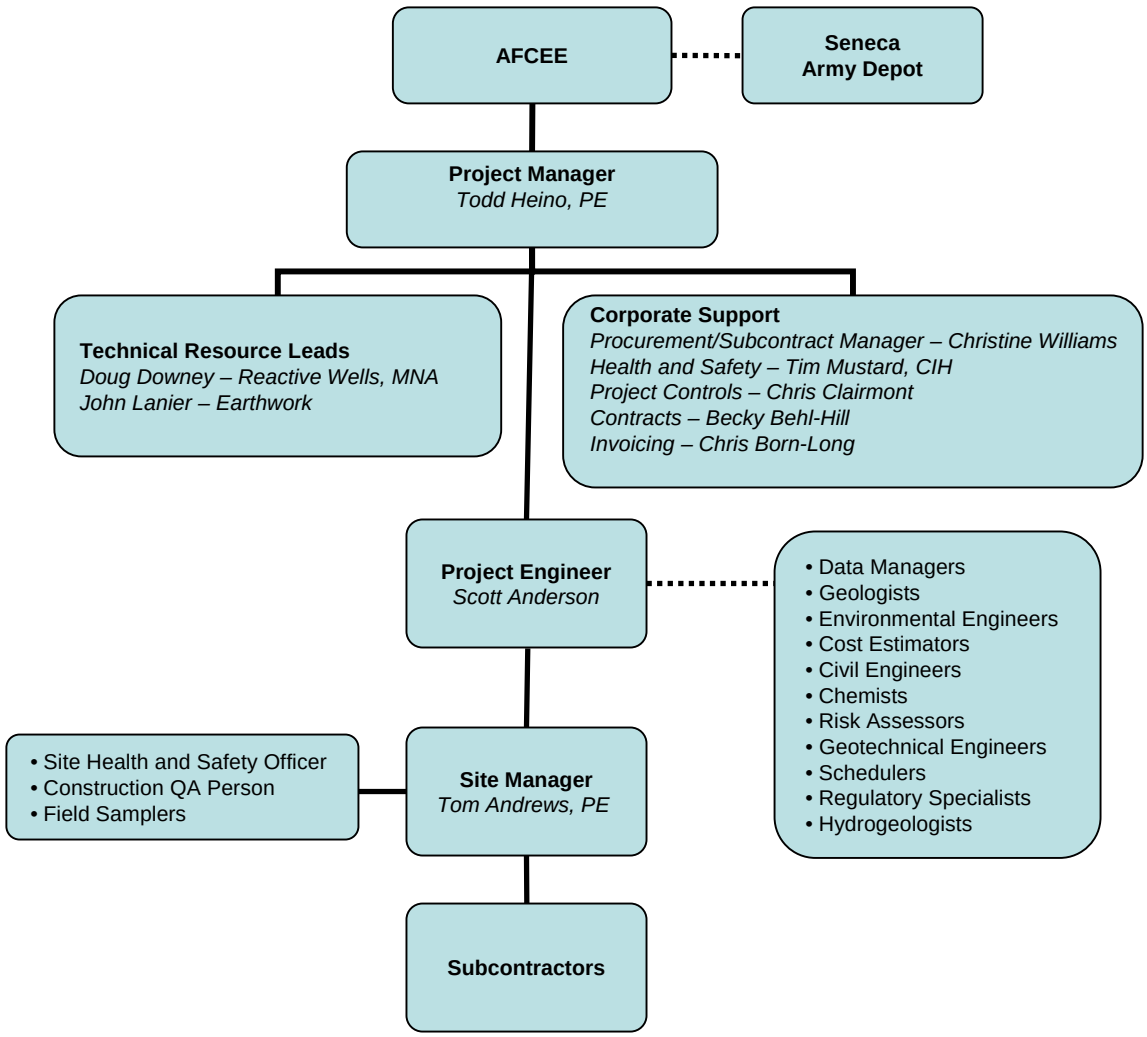
FIGURE 2
SITE LAYOUT

DATE: May 2005

REV:

Figure 3 – Parsons Organization Chart

Seneca Army Depot Activity



Seneca Remediation

Appendix E

Tables

Table 1-A. Potential Chemical-Specific Data Quality Objectives and Preferred Maximum Method Quantitation Limits for Soil/Sediment

Analyte	Abbreviation	CAS #	Human Health Screening Values						Most Stringent Human Health Criteria	Potential ARAR/ TBC*	Ecological Screening Values (Terrestrial)	Eco SV Source	Preferred Maximum Method Quantitation Limit Soil
			Residential Soil (mg/kg)			Industrial Soil (mg/kg)							
			Region IX PRG	Region III RBC	Region VI SSL	Region IX PRG	Region III RBC	Region VI SSL (3)	Soil - Direct Contact (mg/kg)	(mg/kg)	(mg/kg)		(mg/kg)**
VOCs													
Benzene		71-43-2	0.64	12	0.66	1.4	52	1.5	0.64	0.06	0.1	E	**0.06**
1,1-Dichloroethane	DCA	75-34-3	510	16000	590	1700	200000	2100	510	0.2	0.3	E	**0.2**
1,1-Dichloroethene	DCE	75-35-4	120	3900	280	410	51000	430	120	0.4	0.1	I	**0.1**
1,2-Dichloroethane	DCA	107-06-2	0.28	7	0.35	0.6	31	0.77	0.28	0.1	870	E	**0.1**
Cis-1,2-Dichloroethene	cis-DCE	156-59-2	43	780	43	150	10000	150	43	N/A	0.3	E	**0.3**
Trans-1,2-Dichloroethene	trans-DCE	156-60-5	69	1600	63	230	20000	210	63	0.3	0.3	E	**0.3**
Ethyl benzene	EB	100-41-4	400	7800	230	400	100000	230	230	5.5	0.1	E	**0.1**
1,1,1-Trichloroethane	TCA	71-55-6	1200	22000	1400	1200	290000	1400	1200	0.8	29.8	F	**0.8**
Trichloroethene	TCE	79-01-6	0.053	1.6	0.043	0.11	7.2	0.092	0.043	0.7	0.3	E	**0.043**
Toluene	TOL	108-88-3	520	16000	520	520	200000	520	520	1.5	0.1	E	**0.1**
Xylenes		133-02-07	270	16000	210	420	200000	210	210	1.2	0.1	E	**0.1**
Vinyl chloride	VC	75-01-4	0.079	0.09	0.15	0.75	4	0.43	0.079	0.2	0.3	E	**0.079**
SVOCs													
Acenaphthene		83-32-9	3700	4700	3700	29000	61000	33000	3700	50	20	J	**20**
Acenaphthylene		208-96-8	N/A	N/A	N/A	N/A	N/A	N/A	N/A	41	0.1	E	**0.1**
Anthracene		120-12-7	22000	23000	22000	100000	310000	100000	22000	50	0.1	E	**0.1**
Benzo(a)anthracene		56-55-3	0.62	0.87	0.62	2.1	3.9	2.3	0.62	0.224	0.1	E	**0.1**
Benzo(a)pyrene	BaP	50-32-8	0.062	0.087	0.062	0.21	0.39	0.23	0.062	0.061	0.1	E	**0.1**
Benzo(b)fluoranthene		205-99-2	0.62	0.87	0.62	2.1	3.9	2.3	0.62	1.1	0.1	E	**0.1**

Table 1-A. Potential Chemical-Specific Data Quality Objectives and Preferred Maximum Method Quantitation Limits for Soil/Sediment

Analyte	Abbreviation	CAS #	Human Health Screening Values						Most Stringent Human Health Criteria	Potential ARAR/ TBC*	Ecological Screening Values (Terrestrial)	Eco SV Source	Preferred Maximum Method Quantitation Limit Soil
			Residential Soil (mg/kg)			Industrial Soil (mg/kg)							
			Region IX PRG	Region III RBC	Region VI SSL	Region IX PRG	Region III RBC	Region VI SSL (3)	Soil - Direct Contact (mg/kg)	(mg/kg)	(mg/kg)		(mg/kg)**
Benzo(ghi)perylene		191-24-2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	50	0.1	E	**0.1**
Benzo(k)fluoranthene		207-08-9	6.2	8.7	6.2	21	39	23	6.2	1.1	0.1	E	**0.1**
Carbazole		86-74-8	24	32	24	86	140	96	24	N/A	N/A	N/A	**24**
Chrysene		218-01-9	62	87	62	210	390	230	62	0.4	0.1	E	**0.1**
Dibenz(a,h)anthracene		53-70-3	0.062	0.087	0.062	0.21	0.39	0.23	0.062	0.014	0.1	E	**0.1**
Fluoranthene		206-44-0	2300	3100	2300	22000	41000	24000	2300	50	0.1	E	**0.1**
Fluorene		86-73-7	2700	3100	2600	26000	41000	26000	2600	50	30	J	**30**
Indeno(1,2,3-cd)pyrene		193-39-5	0.62	0.87	0.62	2.1	3.9	2.3	0.62	3.2	0.1	E	**0.1**
Naphthalene		91-20-3	56	1600	120	190	20000	190	56	13	0.1	E	**0.1**
Phenanthrene		85-01-8	N/A	N/A	N/A	N/A	N/A	N/A	N/A	50	0.1	E	**0.1**
Pyrene		129-00-0	2300	2300	2300	29000	31000	32000	2300	50	0.1	E	**0.1**
PCB													
Aroclor-1260		11096-82-5	0.22	0.32	0.22	0.74	1.4	0.83	0.22	1.0	0.1	E	**0.1**
Pesticides													
4,4'-DDD		72-54-8	2.4	2.7	2.4	10	12	11	2.4	2.9	0.1	E	**0.1**
4,4'-DDE		72-55-9	1.7	1.9	1.7	7	8.4	7.8	1.7	2.1	0.1	E	**0.1**
4,4'-DDT		50-29-3	1.7	1.9	1.7	7	8.4	7.8	1.7	2.1	0.1	E	**0.1**
Heptachlor epoxide		1024-57-3	0.053	0.07	0.053	0.19	0.31	0.21	0.053	0.02	0.1	E	**0.02**
Explosives													
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4	4.4	5.8	4.4	16	26	17	4.4	N/A	5.8	A	**4.4**

Table 1-A. Potential Chemical-Specific Data Quality Objectives and Preferred Maximum Method Quantitation Limits for Soil/Sediment

Analyte	Abbreviation	CAS #	Human Health Screening Values						Most Stringent Human Health Criteria	Potential ARAR/ TBC*	Ecological Screening Values (Terrestrial)	Eco SV Source	Preferred Maximum Method Quantitation Limit Soil
			Residential Soil (mg/kg)			Industrial Soil (mg/kg)							
			Region IX PRG	Region III RBC	Region VI SSL	Region IX PRG	Region III RBC	Region VI SSL (3)	Soil - Direct Contact (mg/kg)	(mg/kg)	(mg/kg)		(mg/kg)**
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0	3100	3900	3100	31000	51000	34000	3100	N/A	43	H	**43**
2,4,6-Trinitrotoluene (4)	2,4,6-TNT	118-96-7	16	3.9	16	57	51	64	16	N/A	8	B	**8**
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4	1800	2300	1800	18000	31000	21000	1800	N/A	0.38	F	**0.38**
1,3-Dinitrobenzene	1,3-DNB	99-65-0	6.1	7.8	6.1	62	100	68	6.1	N/A	0.66	F	**0.66**
2,4-Dinitrotoluene (1)	2,4-DNT	121-14-2	0.72	0.94	0.72	2.5	4.2	2.8	0.72	N/A	1.28	F	**0.72**
2,6-Dinitrotoluene (1)	2,6-DNT	606-20-2	0.72	0.94	0.72	2.5	4.2	2.8	0.72	1.0	0.033	F	**0.033**
2-Amino-4,6-dinitrotoluene	2-Am-DNT	35572-78-2	12	160	N/A	120	2000	N/A	12	N/A	5.3	H	**5.3**
2-Nitrotoluene	2-NT	88-72-2	0.88	2.8	2.8	2.2	12	14	0.88	N/A	4.1	H	**0.88**
3-Nitrotoluene	3-NT	99-08-1	730	1600	1600	1000	20000	23000	730	N/A	5.3	H	**5.3**
4-Amino-2,6-dinitrotoluene	4-Am-DNT	19406-51-0	12	160	N/A	120	2000	N/A	12	N/A	N/A	N/A	**12**
4-Nitrotoluene	4-NT	99-99-0	12	38	38	30	170	190	12	N/A	9.4	H	**9.4**
Nitrobenzene	NB	98-95-3	20	39	20	100	510	110	20	0.2	40	C	**0.2**
Nitroglycerin	NG	55-63-0	35	46	N/A	120	200	N/A	35	N/A	150	H	**35**
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-45-8	610	310	240	6200	4100	2700	240	N/A	2	H	**2**
Pentaerythritol Tetranitrate	PETN	78-11-5	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	21000	H	**21000**
Inorganics													
Aluminum	Al	7429-90-5	76000	78000	76000	100000	1000000	100000	76000	19300	50	C	**50**
Antimony	Sb	7440-36-0	31	31	31	410	410	450	31	5.9	0.27	A	**0.27**

Table 1-A. Potential Chemical-Specific Data Quality Objectives and Preferred Maximum Method Quantitation Limits for Soil/Sediment

Analyte	Abbreviation	CAS #	Human Health Screening Values						Most Stringent Human Health Criteria	Potential ARAR/ TBC*	Ecological Screening Values (Terrestrial)	Eco SV Source	Preferred Maximum Method Quantitation Limit Soil
			Residential Soil (mg/kg)			Industrial Soil (mg/kg)							
			Region IX PRG	Region III RBC	Region VI SSL	Region IX PRG	Region III RBC	Region VI SSL (3)	Soil - Direct Contact (mg/kg)	(mg/kg)	(mg/kg)		(mg/kg)**
Arsenic	As	7440-38-2	0.39	0.43	0.39	1.6	1.9	1.8	0.39	8.2	18	A	**0.39**
Barium	Ba	7440-38-2	5400	5500	5500	67000	72000	79000	5400	300	330	A	**300**
Beryllium	Be	7440-41-7	150	160	150	1900	2000	2200	150	1.1	21	C	**1.1**
Cadmium	Cd	7440-43-9	37	39	39	450	510	560	37	2.3	0.36	C	**0.36**
Calcium	Ca	7440-70-2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	121000	N/A	N/A	**121000**
Chromium (2)	Cr	7440-47-3	210	230	210	450	3100	450	210	29.6	26	A	**26**
Cobalt	Co	7440-48-4	900	1600	900	1900	20000	1900	900	30	13	A	**13**
Copper	Cu	7440-50-8	3100	3100	2900	41000	41000	42000	2900	33	61	A	**33**
Iron	Fe	7439-89-6	23000	23000	23000	100000	310000	100000	23000	36500	N/A	N/A	**23000**
Lead	Pb	7439-92-1	400	N/A	400	800	N/A	800	400	24.8	11	A	**11**
Magnesium	Mg	7439-95-4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	21500	4400	E	**4400**
Manganese	Mn	7439-96-5	1800	1600	3200	19000	20000	35000	1600	1060	152	A	**152**
Molybdenum	Mo	7439-98-7	390	390	390	5100	5100	5700	390	N/A	0.59	E	**0.59**
Nickel	Ni	7440-02-0	1600	1600	1600	20000	20000	23000	1600	49	38	A	**38**
Potassium	K	7440-09-7	N/A	N/A	N/A	N/A	N/A	N/A	N/A	2380	N/A	N/A	**2380**
Selenium	Se	7782-49-2	390	390	390	5100	5100	5700	390	2	0.50	A	**0.50**
Silver	Ag	7440-22-4	390	390	390	5100	5100	5700	390	0.75	2.0	C	**0.75**
Sodium	Na	7440-23-5	N/A	N/A	N/A	N/A	N/A	N/A	N/A	172	N/A	N/A	**172**
Strontium	Sr	7440-24-6	47000	47000	47000	100000	610000	100000	47000	N/A	N/A	N/A	**47000**
Thallium	Tl	7440-28-0	5.2	5.5	N/A	67	72	N/A	5.2	0.7	1.0	C	**0.7**

Table 1-A. Potential Chemical-Specific Data Quality Objectives and Preferred Maximum Method Quantitation Limits for Soil/Sediment

Analyte	Abbreviation	CAS #	Human Health Screening Values						Most Stringent Human Health Criteria	Potential ARAR/ TBC*	Ecological Screening Values (Terrestrial)	Eco SV Source	Preferred Maximum Method Quantitation Limit Soil
			Residential Soil (mg/kg)			Industrial Soil (mg/kg)							
			Region IX PRG	Region III RBC	Region VI SSL	Region IX PRG	Region III RBC	Region VI SSL (3)	Soil - Direct Contact (mg/kg)	(mg/kg)	(mg/kg)		(mg/kg)**
Titanium	Ti	7440-32-6	100000	310000	N/A	100000	4100000	N/A	100000	N/A	N/A	N/A	**100000**
Vanadium	V	7440-62-2	78	78	78	1000	1000	1100	78	150	2.0	C	**2**
Zinc	Zn	7440-66-6	23000	23000	23000	100000	310000	100000	23000	110	120	A	**110**
Zirconium	Zr	7440-67-7	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	**N/A**
Mercury	Hg	7439-97-6	23	23	23	310	310	340	23	0.1	0.10	C	**0.1**
Phosphorus (White)	WP or P4	7723-14-0	1.6	1.6	1.6	20	20	23	1.6	N/A	N/A	N/A	**1.6**
Perchlorate	ClO4	14797-73-0	7.8	55	7.8	100	720	110	7.8	N/A	N/A	N/A	**7.8**

* Potential ARAR/TBC values are from NYSDEC Technical and Administrative Guidance Memorandum #4046 (on-line resources available at http://www.dec.state.ny.us/website/der/tagms/prtg4046.html)

** If laboratory cannot meet any of the preferred QLs with routine SW846 methodology (as supported by MDLs that are no greater than 1/3 QL), laboratory's QL must be identified in Laboratory submittal as failing to meet the QL. Some screening values cannot be obtained with routine methodology to the QL. In those cases, the QL achievable with a routine SW846 methodology would be accepted.

(1) Carcinogenic DNT mixture values used if more conservative than noncarcinogenic isomer-specific values
(2) Total chromium values used if available. All Region III values are based on hexavalent chromium.
(3) Lower of the industrial values provided (industrial w/o dermal vs. industrial/outdoor)
(4) Noncancer RBCs at an HI of 0.1 provided because screening at an HI of 0.1, in accordance with Region III guidance, will result in noncancer RBCs being lower than the cancer RBCs

Region IX PRGs, dated January, 2005
Region III RBCs, dated April 2005
Region VI SSLs, dated 21 December 2004

Eco Screening Value Sources:

A USEPA EcoSSLs
B Los Alamos Nuclear Lab Screening Level
C USEPA Region IV Eco Screening Values
D San Francisco Regional Water Quality Control Board Surface Water Screening Values
E USEPA Region III Freshwater Screening Benchmarks
F USEPA Region V Ecological Data Quality Levels
G Talmage, et. al. 1999
H Los Alamos National Laboratory (LANL), ECORISK Database, 2004
I. CCME, 2003
J. Oak Ridge, 1997

Table 1-B. Potential Chemical-Specific Data Quality Objectives and Preferred Maximum Method Quantitation Limits for Groundwater/Surface Water

			Human Health Screening Values										
			Tap Water (ug/L)			Federal Drinking Water Criteria (ug/L)		Federal Ambient Water Quality (ug/L)		NYSDEC GA Standards (ug/L)*	Ecological Screening Values (ug/L)	Eco SV Source	Preferred Maximum Method Quantitation Limit Aqueous (ug/L)**
Analyte	Abbreviation	CAS #	Region IX PRG	Region III RBC	Region VI SSL	MCLs	HA	CMC	CCC				
VOCs													
Benzene		71-43-2	0.35	0.34	0.35	5	100(6)	N/A	N/A	1	5300	E	0.34
1,1-Dichloroethane	DCA	75-34-3	810	900	810	N/A	N/A	N/A	N/A	5	160000	E	5
1,1-Dichloroethene	DCE	75-35-4	340	350	340	7	6(6)	N/A	N/A	5	11600	E	5
1,2-Dichloroethane	DCA	107-06-2	0.12	0.12	0.12	5	40(6)	N/A	N/A	0.6	20000	E	0.12
Cis-1,2-Dichloroethene	cis-DCE	156-59-2	61	61	61	70	70	N/A	N/A	5	11600	E	5
Trans-1,2-Dichloroethene	trans-DCE	156-60-5	120	110	120	100	100	N/A	N/A	5	11600	E	5
Ethyl benzene	EB	100-41-4	1300	1300	1300	700	700	N/A	N/A	5	32000	E	5
1,1,1-Trichloroethane	TCA	71-55-6	3200	3200	840	200	200	N/A	N/A	5	76	F	5
Trichloroethene	TCE	79-01-6	0.028	0.026	0.028	5	300(6)	N/A	N/A	5	21900	E	0.026
Toluene	TOL	108-88-3	720	750	720	1000	1000	N/A	N/A	5	17000	E	5
Xylenes		133-02-07	210	210	200	10000	7000(7)	N/A	N/A	5	6000	E	5
Vinyl chloride	VC	75-01-4	0.02	0.015	0.043	2	2(6)	N/A	N/A	2	11600	E	0.02
SVOCs													
Acenaphthene		83-32-9	370	370	370	N/A	2000(7)	N/A	N/A	20	520	E	20
Acenaphthylene		208-96-8	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	4840	F	4840
Anthracene		120-12-7	1800	1800	1800	N/A	10000(7)	N/A	N/A	50	0.1	E	0.1
Benzo(a)anthracene		56-55-3	0.092	0.092	0.092	N/A	N/A	N/A	N/A	0.002	6.3	E	0.002
Benzo(a)pyrene	BaP	50-32-8	0.0092	0.0092	0.0092	0.2	0.5(6)	N/A	N/A	ND	0.014	F	0.0092
Benzo(b)fluoranthene		205-99-2	0.092	0.092	0.092	N/A	N/A	N/A	N/A	0.002	9.07	F	0.002
Benzo(ghi)perylene		191-24-2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	7.64	F	7.64

Table 1-B. Potential Chemical-Specific Data Quality Objectives and Preferred Maximum Method Quantitation Limits for Groundwater/Surface Water

			Human Health Screening Values										
			Tap Water (ug/L)			Federal Drinking Water Criteria (ug/L)		Federal Ambient Water Quality (ug/L)		NYSDEC GA Standards (ug/L)*	Ecological Screening Values (ug/L)	Eco SV Source	Preferred Maximum Method Quantitation Limit Aqueous (ug/L)**
Analyte	**Abbreviation**	**CAS #**	**Region IX PRG**	**Region III RBC**	**Region VI SSL**	**MCLs**	**HA**	**CMC**	**CCC**				
Benzo(k)fluoranthene		207-08-9	0.92	0.92	0.92	N/A	N/A	N/A	N/A	0.002	0.05	H	0.002
Carbazole		86-74-8	3.4	3.3	3.4	N/A	N/A	N/A	N/A	N/A			3.3
Chrysene		218-01-9	9.2	9.2	9.2	N/A	N/A	N/A	N/A	N/A			9.2
Dibenz(a,h)anthracene		53-70-3	0.0092	0.0092	0.0092	N/A	N/A	N/A	N/A	N/A			0.0092
Fluoranthene		206-44-0	1500	1500	1500	N/A	N/A	N/A	N/A	50	3980	E	50
Fluorene		86-73-7	240	240	240	N/A	1000(7)	N/A	N/A	50	430	E	50
Indeno(1,2,3-cd)pyrene		193-39-5	0.092	0.092	0.092	N/A	N/A	N/A	N/A	0.002	4.31	F	0.002
Naphthalene		91-20-3	6.2	6.5	6.2	N/A	100	N/A	N/A	10	100	E	6.2
Phenanthrene		85-01-8	N/A	N/A	N/A	N/A	N/A	N/A	N/A	50	6.3	E	6.3
Pyrene		129-00-0	180	180	180	N/A	N/A	N/A	N/A	50	0.3	F	0.3
PCB													
Aroclor-1260		11096-82-5	0.034	0.033	0.034	0.5	10(6)	N/A	0.014	5	0.014	E	0.014
Pesticides													
4,4'-DDD		72-54-8	0.28	0.28	0.28	N/A	N/A	N/A	N/A	0.3	0.6	E	0.28
4,4'-DDE		72-55-9	0.2	0.2	0.2	N/A	N/A	N/A	N/A	0.2	1050	E	0.2
4,4'-DDT		50-29-3	0.2	0.2	0.2	N/A	N/A	N/A	N/A	0.2	0.001	E	0.2
Heptachlor epoxide		1024-57-3	0.0074	0.0074	0.0074	0.2	0.4(6,7)	0.52	0.0038	0.03	0.0038	E	0.0038
Explosives													
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4	0.61	0.61	0.61	N/A	2	N/A	N/A	5	360	E	0.61
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0	1800	1800	1800	N/A	400	N/A	N/A	N/A	150	E	150
2,4,6-Trinitrotoluene (4)	2,4,6-TNT	118-96-7	2.2	1.8	2.2	N/A	2	N/A	N/A	5	100	E	1.8

Table 1-B. Potential Chemical-Specific Data Quality Objectives and Preferred Maximum Method Quantitation Limits for Groundwater/Surface Water

			Human Health Screening Values										
			Tap Water (ug/L)			Federal Drinking Water Criteria (ug/L)		Federal Ambient Water Quality (ug/L)		NYSDEC GA Standards (ug/L)*	Ecological Screening Values (ug/L)	Eco SV Source	Preferred Maximum Method Quantitation Limit Aqueous (ug/L)**
Analyte	**Abbreviation**	**CAS #**	**Region IX PRG**	**Region III RBC**	**Region VI SSL**	**MCLs**	**HA**	**CMC**	**CCC**				
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4	1100	1100	1100	N/A	N/A	N/A	N/A	5	11	G	5
1,3-Dinitrobenzene	1,3-DNB	99-65-0	3.6	3.7	3.7	N/A	1	N/A	N/A	5	20	G	1
2,4-Dinitrotoluene (1)	2,4-DNT	121-14-2	0.099	0.098	0.099	N/A	5 (6)	N/A	N/A	5	310	C	0.099
2,6-Dinitrotoluene (1)	2,6-DNT	606-20-2	0.099	0.098	0.099	N/A	5 (6)	N/A	N/A	5	81	E	0.098
2-Amino-4,6-dinitrotoluene	2-Am-DNT	35572-78-2	7.3	7.3	N/A	N/A	N/A	N/A	N/A	5	20	G	5
2-Nitrotoluene	2-NT	88-72-2	0.049	0.046	0.29	N/A	N/A	N/A	N/A	5	N/A		0.046
3-Nitrotoluene	3-NT	99-08-1	120	120	120	N/A	N/A	N/A	N/A	5	750	E	5
4-Amino-2,6-dinitrotoluene	4-Am-DNT	19406-51-0	7.3	7.3	N/A	N/A	N/A	N/A	N/A	5	N/A		5
4-Nitrotoluene	4-NT	99-99-0	0.66	0.62	4.0	N/A	N/A	N/A	N/A	5	1900	E	0.62
Nitrobenzene	NB	98-95-3	3.4	3.5	3.4	N/A	N/A	N/A	N/A	0.4		270	0.4
Nitroglycerin	NG	55-63-0	4.8	4.8	N/A	N/A	5	N/A	N/A	N/A	138	E	4.8
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-45-8	360	150	150	N/A	N/A	N/A	N/A	5	5800	H	5
Pentaerythritol Tetranitrate	PETN	78-11-5	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	85000	E	85000
Inorganics													
Aluminum	Al	7429-90-5	36000	37000	37000	50 (5)	N/A	750(9)	87(9)	N/A	25	E	25
Antimony	Sb	7440-36-0	15	15	15	6	6	N/A	N/A	3	6.0	D	3
Arsenic	As	7440-38-2	0.045	0.045	0.045	10	10(7)	340	150	25	0.14	D	0.045
Barium	Ba	7440-38-2	2600	2600	2600	2000	2000	N/A	N/A	1000	1000	D	1000
Beryllium	Be	7440-41-7	73	73	73	4	70(7)	N/A	N/A	3	2.7	D	2.7
Cadmium	Cd	7440-43-9	18	18	18	5	5	2.0	0.25	5	2.2	D	0.25
Calcium	Ca	7440-70-2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Chromium (2)	Cr	7440-47-3	110	110	110	100	100(7)	16	11	50	50	D	11

Table 1-B. Potential Chemical-Specific Data Quality Objectives and Preferred Maximum Method Quantitation Limits for Groundwater/Surface Water

			Human Health Screening Values										
			Tap Water (ug/L)			Federal Drinking Water Criteria (ug/L)		Federal Ambient Water Quality (ug/L)		NYSDEC GA Standards (ug/L)*	Ecological Screening Values (ug/L)	Eco SV Source	Preferred Maximum Method Quantitation Limit Aqueous (ug/L)**
Analyte	Abbreviation	CAS #	Region IX PRG	Region III RBC	Region VI SSL	MCLs	HA	CMC	CCC				
Cobalt	Co	7440-48-4	730	730	730	N/A	N/A	N/A	N/A	N/A	3.0	D	3
Copper	Cu	7440-50-8	1500	1500	1400	1300 1000 (5)	N/A	13	9.0	200	9.0	D	9
Iron	Fe	7439-89-6	11000	11000	11000	300 (5)	N/A	N/A	1000(9)	300	320	E	300
Lead	Pb	7439-92-1	N/A	N/A	15	15	N/A	65	2.5	25	2.5	D	2.5
Magnesium	Mg	7439-95-4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	35000	N/A	N/A	35000
Manganese	Mn	7439-96-5	880	730	1700	50 (5)	300	N/A	N/A	300	14500	E	50
Mercury	Hg	7439-97-6	11	11	11	2	2	1.4	0.77	0.7	0.77	D	0.7
Molybdenum	Mo	7439-98-7	180	180	180	N/A	40	N/A	N/A	N/A	N/A	N/A	40
Nickel	Ni	7440-02-0	730	730	730	N/A	100	470	52	100	52	D	52
Potassium	K	7440-09-7	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Selenium	Se	7782-49-2	180	180	180	50	50	N/A	5.0	10	5.0	D	5
Silver	Ag	7440-22-4	180	180	180	100 (5)	100	3.2	N/A	50	0.34	D	0.34
Sodium	Na	7440-23-5	N/A	N/A	N/A	20000 (8)	N/A	N/A	N/A	20000	N/A	N/A	20000
Strontium	Sr	7440-24-6	22000	22000	22000	N/A	4000	N/A	N/A	N/A	N/A	N/A	4000
Thallium	Tl	7440-28-0	2.4	2.6	2.9	2	0.5	N/A	N/A	0.5	2.0	D	0.5
Titanium	Ti	7440-32-6	150000	150000	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	150000
Vanadium	V	7440-62-2	36	37	37	N/A	N/A	N/A	N/A	N/A	19	D	19
Zinc	Zn	7440-66-6	11000	11000	11000	5000 (5)	2000	120	120	2000	120	D	120
Zirconium	Zr	7440-67-7	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Phosphorus (White)	WP or P4	7723-14-0	0.73	0.73	0.73	N/A	0.1	N/A	N/A	N/A	0.1	E	0.1
Perchlorate	ClO4	14797-73-0	3.6	26	3.7	N/A	N/A	N/A	N/A	N/A	N/A	N/A	3.6

* New York State Ambient Water Quality Standards, GA (http://www.dec.state.ny.us/website/regs/part701.html)
** If laboratory cannot meet any of these QLs with routine SW846 methodology (as supported by MDLs that are no greater than 1/3 QL), laboratory's QL must be identified in Laboratory submittal as failing to meet the QL. Some screening values cannot be obtained with routine methodology to the QL.

(1) Carcinogenic DNT mixture values used if more conservative than noncarcinogenic isomer-specific values
(2) Total chromium values used if available. All Region III values are based on hexavalent chromium.
(3) Lower of the industrial values provided (industrial w/o dermal vs. industrial/outdoor)
(4) Noncancer RBCs at an HI of 0.1 provided because screening at an HI of 0.1, in accordance with Region III guidance, will result in noncancer RBCs being lower than the cancer RBCs
(5) All MCLs are primary except those with this footnote.
(6) All HAs are lifetime except those footnoted, which are based on 10-4 cancer risk
(7) Drinking Water Equivalent Level
(8) Drinking Water Advisory

Sources:
A USEPA EcoSSLs
B Los Alamos Nuclear Lab Screening Level
C USEPA Region IV Eco Screening Values
D San Francisco Regional Water Quality Control Board Surface Water Screening Values
E USEPA Region III Freshwater Screening Benchmarks
F USEPA Region V Ecological Data Quality Levels
G Talmage, et. al. 1999
H. Dutch, 2000.

Region IX PRGs, dated January 2005

Region VI SSLs, dated 21 December 2004

Region III RBCs, dated April 2005

Table 2
Project Communication Pathways

Communication Drivers	Responsible party	Name	Phone Number	Procedure
Approval of QAPP/amendments to QAPP	USEPA PM NYSDEC PM SEDA PM	Julio F. Vazquez Kuldeep K. Gupta Steve Absolom	212-637-4323 518-402-9620 607-869-1309	Parsons PM will initiate calls to SEDA if review time passed the scheduled review period. SEDA will then call USEPA and NYSDCE PM to discuss QAPP/amendment schedule.
Notification of delays or changes to field work	Parsons PM	Todd Heino	617-449-1405	Parsons field team leader will update PM daily filed progress. Parsons PM will update SEDA any delay or change of field activities.
Recommendations to stop work and initiation of corrective action	Parsons PM Parsons Health and Safety Officer	Todd Heino	617-449-1405	Parsons PM or Health and Safety Officer will initiate a work stoppage due to QA/QC concerns, health and safety concerns, or any other project related concerns.
Reporting of issues related to analytical data quality	Parsons Chemist	Katherine Lapierre	512-719-6000x6806	Parsons chemist will initiate discussion with the Laboratory with any data quality issues.
Quality Assurance and Changes to the QAPP	PARSONS Quality Assurance Officer	James Lowerre	617-449-1559	Parsons QA Officer will initiate internal discussion with PM and project team regarding any QA issues and corrective actions.

Table 3
Summary of Screening Analytical Methods and Method Detection Limits

Reference Number	Title, Revision Date, and/or Number	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)	Method Detection Limits [1]
USEPA Method 160.3	Residue, Total (Gravimetric, Dried at 103-105°C) Approved for NPDES (Issued 1971)	Percent Solids	Drying Oven	Laboratory	N	10 mg/L
SW846 Method 1010A/ 1020B/ 1030	Pensky-Martens Closed-Cup Method For Determining Ignitability, Revision 1, 2002; Small Scale Closed-Cup Method For Determining Ignitability, Revision 2, 2002; Ignitability of Solids, Revision 0, 1996	Ignitability	Pensky-Martens Closed-Cup Tester; Small Scale Closed-Cup Apparatus; A Bunsen burner	Laboratory	No	
SW846 Method 1110	Corrosivity Toward Steel, Revision 0, 1996	Corrosivity	A resin flask	Laboratory	No	
SW846 Method 9040C	Electrometric Measurement, Revision 3, 2002	pH (water)	pH meter	Laboratory/Field	No	
SW846 Method 9045D	Soil and Waste pH, Revision 4, 2002	pH (soil)	pH meter	Laboratory/Field	No	
SW846 Method 9050A	Specific Conductance, Revision 1, 1996	conductance	Self-contained conductivity instruments	Laboratory/Field	No	
SW846 Method 9060A/USEPA Method 415.1/Lloyd Kahn	Total Organic Carbon, Revision 1, 2002 Organic Carbon, Total (Combustion Or Oxidation) - Approved for NPDES (Editorial Revision 1974) Determination of Total Organic Carbon in Sediment (Lloyd Kahn Method), Lloyd Kahn, 1988	Total Organic Carbon	carbonaceous analyzer	Laboratory	No	1 mg/L
USEPA Method 130.1	Hardness, Total (mg/L as $CaCO_3$) (Colorimetric, Automated EDTA) - Approved for NPDES (Issued 1971)	Hardness	Spectrophotometer	Laboratory/Field	No	10 mg/L
USEPA Method 170.1	Temperature - Approved for NPDES (Issued 1974)	Temperature	Thermometer	Laboratory/Field	No	
USEPA Method 180.1	Determination Of Turbidity By Nephelometry, Revision 2, 1993.	Turbidity	Nephelometry	Laboratory/Field	No	
E310.1/Hach	Alkalinity (Titrimetric, pH 4.5) - Approved	Alkalinity	PH meter or electrically	Laboratory/Field	No	10 mg/L

Reference Number	Title, Revision Date, and/or Number	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)	Method Detection Limits [1]
Method 8203 or similar	for NPDES (Editorial Revision 1978)		operated titrator			
E360.1 with commercially available probe	Oxygen, Dissolved (Membrane Electrode) - Approved for NPDES (Issued 1971)	Dissolved oxygen	Oxygen Analyzer	Laboratory/Field	No	
Organic Vapor Analysis	SOP (Section 16 of this SAP)	Hydrocarbon vapor	Photoionization Detector (PID)	Field	No	0.05~0.5 ug/L or ug/kg
USEPA Method 410.1	Chemical Oxygen Demand (Titrimetric, Mid-Level) - Approved for NPDES (Editorial Revision 1978)	Chemical Oxygen Demand		Laboratory	No	50 mg/L
USEPA 351.2 / SM4500	Determination of Total Kjeldahl Nitrogen by Semi-Automated Colorimetry	Total Kjeldahl Nitrogen (TKN)	Colorimeter	Laboratory	N	
USEPA Method 300.1/ SW846 Method 9056	Determination Of Inorganic Anions In Drinking Water By Ion Chromatography, Revision 1. Determination Of Inorganic Anions By Ion Chromatography, Revision 0, 1994	Nitrate, nitrite, chloride, sulfate	Ion Chromatography	Laboratory	No	Nitrate-N: 0.008 mg/L; Nitrite-N: 0.001 mg/L; Chloride: 0.004 mg/L Sulfate: 0.019 mg/L
USEPA Method 353.1	Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Hydrazine Reduction) - Approved for NPDES and SDWA (Reissued w/ Rev. 1978)	Nitrate, nitrite	Spectrophotometer	Laboratory	No	0.01 mg/L
ASTM D1498 with commercially available ORP	Standard Practice for Oxidation-Reduction Potential of Water	Oxidation-reduction potential	ORP instrument	Field	No	
Hach 8146	Iron, Ferrous Method 8146 DR/2500.	Ferrous iron	Hach system	Field	No	0.02 mg/L
Hach 8034	Manganese Method 8034 WAH, DR/4000, DR/2500, DR/2400, or Genesys	Manganese	Hach system	Field	N	0.2 mg/L
Hach 8131	Sulfide Method 8131 WAH, DR/4000, DR/2500, DR/2400, or Genesys	Sulfide	Hach system	Field	N	5 mg/L
Hach 8205	Carbon Dioxide Method 8205 WAH	CO_2	Hach system	Field	N	10 mg/L

Note: 1. Method detection limit listed by the method. Method detection limit provided by the laboratory will be reviewed for each specific project.

Table 4
Summary of Definitive Analytical Methods

Reference Number	Title, Revision Date, and/or Number	Analytical Group	Instrument
SW846 Method 8260B	Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, 1996	Volatile organic compounds	GC/MS
SW846 Method 8270C	Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, 1998	Semivolatile Organic Compounds	GC/MS
SW846 Method 8081B	Organochlorine Pesticides By Gas Chromatography, Draft Revision 2A, 1999	Organochlorine Pesticides	GC
SW846 Method 8082A	Polychlorinated Biphenyls (PCBs) By Gas Chromatography, Draft Revision 1A, 1999	PCBs	GC
SW846 Method 8330	Nitroaromatics And Nitramines By High Performance Liquid Chromatography (HPLC), Revision 0, 1994	Nitroaromatics And Nitramines	HPLC
USEPA Method 524.2	Measurement Of Purgeable Organic Compounds In Water By Capillary Column Gas Chromatography/Mass Spectrometry, Revision 4.1, 1995	Volatile organic compounds	GC/MS
SW846 Method 6010B	Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 2, 1996	Metals	ICP/AS
SW846 Method 6020	Inductively Coupled Plasma - Mass Spectrometry, Revision 0, 1994	Metals	ICP/MS
SW846 Method 7470A	Mercury In Liquid Waste (Manual Cold-Vapor Technique), Revision 1, 1994	Mercury	Atomic absorption spectrophotometer or equivalent
SW846 Method 7471A	Mercury In Solid Or Semisolid Waste (Manual Cold-Vapor Technique), Revision 2, 1998	Mercury	Atomic absorption spectrophotometer or equivalent
SW846 Method 7580A	White Phosphorus (P) By Solvent Extraction And Gas Chromatography, Revision 0, 1990	White Phosphorus	Gas chromatograph
USEPA Method 314.0	Determination Of Perchlorate In Drinking Water Using Ion Chromatography, Revision 1.0, 1999	Perchlorate	Ion Chromatograph
SW846 Method 7196A	Chromium, Hexavalent (Colorimetric), Revision 1, 1992	Hexavalent chromium	Spectrophotometer or filter photometer
RSK-175 and USEPA Method 8015D	Analysis of Dissolved Methane, Ethane, and Ethylene in Groundwater by a Standard Gas Chromatograph Technique Nonhalogenated Organics Using GC/FID, Revision 4, 2003	Methane, Ethane, Ethene	Gas chromatograph/flame ionization detector

Notes:

1) The above reference methods are from the following literatures:
   a) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (USEPA SW-846, Third Edition) and its subsequent updates.
   b) Methods for Chemical Analysis of Water and Waste (USEPA, 1979).
2) All definitive analyses will be conducted by selected laboratory. The laboratory should conduct the analyses in accordance with all NYSDEC ASP requirements and requirements specified in this ASP.

Table 5-A

Sample Containers, Preservatives, and Holding Times for Soils and Sediments

Parameter	Sample Container	Preservative	Technical Holding Time
Metals	1 4 oz wide-mouth glass w/ Teflon-lined cap	Ice, Cool to 4°C	28 days (Hg); 24 hours (hex chromium); 180 days (others)
Explosives	1 4 oz wide-mouth glass w/ Teflon-lined cap	Ice, Cool to 4°C	14/40 days[a]
Perchlorate	1 4 oz wide-mouth glass w/ Teflon-lined cap	Ice, Cool to 4°C	28 days
pH	1 4 oz wide-mouth glass w/ Teflon-lined cap	Ice, Cool to 4°C	ASAP
SVOCs	1 4 oz wide-mouth glass w/ Teflon-lined cap	Ice, Cool to 4°C	14/40 days[a]
Pesticides/PCBs	1 4 oz wide-mouth glass w/ Teflon-lined cap	Ice, Cool to 4°C	14/40 days[a]
VOCs	1 4 oz wide-mouth glass w/ Teflon-lined cap	Ice, Cool to 4°C (low level sample) Methanol, Ice, Cool to 4°C (medium level sample)	10 days
TOC	1 4 oz wide-mouth glass w/ Teflon-lined cap	Ice, Cool to 4°C	ASAP
TCLP VOC	1 8 oz wide mouth glass with Teflon-lined cap	Ice, Cool to 4°C	14/NA/14[b]
TCLP SVOC	1 8 oz wide mouth glass with Teflon-lined cap	Ice, Cool to 4°C	14/7/40 [b]
TCLP Mercury	1 8 oz wide mouth glass with Teflon-lined cap	Ice, Cool to 4°C	28/NA/28 (mercury) [b]
TCLP Metals (except Mercury)	1 8 oz wide mouth glass with Teflon-lined cap	Ice, Cool to 4°C	180/NA/180 [b]

Notes:

[a] number of days between sample collection and extraction / number of days between extraction and analysis;

[b] number of days between sample collection and TCLP extraction/number of days between TCLP extraction and preparative extraction/number of days between preparative extraction and analysis.

Table 5-B

Sample Containers, Preservatives, and Holding Times for Aqueous Samples

Parameter	Sample Container	Preservative	Technical Holding Time
Metals	1 500-ml plastic bottle	pH<2, with HNO3, Ice, Cool to 4ºC	28 days (Hg); 24 hours (hex chromium); 180 days
Explosives	2 1-L amber bottles	Ice, Cool to 4°C	7/40 days[a]
Inorganic Ions	1 500-ml plastic bottle	Ice, Cool to 4°C	Nitrate, Nitrite, Phosphate – 24 hours All others – 28 days
Perchlorate	1 250-ml plastic or glass bottle	Ice, Cool to 4°C	28 days
Hardness	1 100-ml plastic bottle	pH<2, with HNO3, Ice, Cool to 4ºC	6 months
SVOCs	2 1-L amber bottle	Ice, Cool to 4°C	7/40 days[a]
Turbidity	1 500-ml plastic bottle	Ice, Cool to 4°C	24 hours
TOC/COD	1 1-L amber bottle	Ice, Cool to 4°C, with H_2SO_4 to pH<2	28 days
Pesticides	1 1-L amber bottle	Ice, Cool to 4°C	7/40 days[a]
PCBs	1 1-L amber bottle	Ice, Cool to 4°C	7/40 days[a]
VOCs	3 40 mL VOA vials	pH<2, with HCl, Ice ,Cool to 4°C	10 days[a] (7 days if unpreserved)
TKN	1 1-L amber bottle	Ice, Cool to 4°C, with H_2SO_4 to pH<2	14 days
MEE	1 60-ml serum bottle w/ Teflon-lined cap	Ice, Cool to 4°C	14 days[a]
TCLP VOC	3 40 mL VOA vials	Ice, Cool to 4°C	14/NA/14[b]
TCLP SVOC [c]	1 1-L amber bottle	Ice, Cool to 4°C	14/7/40 [b]
TCLP Mercury	1 1-L amber bottle	Ice, Cool to 4°C	28/NA/28 [b]
TCLP Metals (except Mercury)	1 1-L amber bottle	Ice, Cool to 4°C	180/NA/180 [b]

Notes:

[a] number of days between sample collection and extraction / number of days between extraction and analysis;

[b] number of days between sample collection and TCLP extraction/number of days between TCLP extraction and preparative extraction/number of days between preparative extraction and analysis.

[c] TCLP SVOCs includes all semivolatiles, pesticides, and herbicides.

Table 6-A

Target Analyte List for Volatile Organic Compounds by GC/MS

(Based on NYSDEC ASP Requirement for Superfund CLP Program)

Volatile Organic Compound	CAS #	Quantitation Limits – CLP Low Concentration TCL ug/L	Quantitation Limits – CLP TCL		
			Water ug/L	Low Soil ug/kg	Med Soil ug/kg
Dichlorodifluoromethane	75-71-8	--	10	10	1200
Chloromethane	74-87-3	1	10	10	1200
Bromomethane	74-83-9	1	10	10	1200
Vinyl chloride	75-01-4	1	10	10	1200
Chloroethane	75-00-3	1	10	10	1200
Trichlorofluoromethane	75-69-4	--	10	10	1200
1,1-Dichloroethene	75-35-4	1	10	10	1200
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	--	10	10	1200
Acetone	67-64-1	5	10	10	1200
Carbon Disulfide	75-15-0	1	10	10	1200
Methyl Acetate	79-20-9	--	10	10	1200
Methylene chloride	75-09-2	2	10	10	1200
trans-1,2-Dichloroethene	156-60-5	1	10	10	1200
Methyl tert-Butyl Ether	1634-04-4	--	10	10	1200
1,1-Dichloroethane	75-35-3	1	10	10	1200
Cis-1,2-Dichloroethene	156-59-2	1	10	10	1200
2-Butanone	78-93-3	5	10	10	1200
Chloroform	67-66-3	1	10	10	1200
1,1,1-Trichloroethane	71-55-6	1	10	10	1200
Cyclohexane	110-82-7	--	10	10	1200
Carbon tetrachloride	56-23-5	1	10	10	1200
Benzene	71-43-2	1	10	10	1200
1,2-Dichloroethane	107-06-2	1	10	10	1200
Trichloroethene	79-01-6	1	10	10	1200
Methylcyclohexane	108-87-2	--	10	10	1200
1,2-Dichloropropane	78-87-5	1	10	10	1200
Bromodichloromethane	75-27-4	1	10	10	1200
Cis-1,3-Dichloropropene	10061-01-5	1	10	10	1200
4-Methyl-2-pentanone	108-10-1	5	10	10	1200
Toluene	108-88-3	1	10	10	1200
trans-1,3-Dichloropropene	10061-02-6	1	10	10	1200
1,1,2-Trichloroethane	79-00-5	1	10	10	1200
Tetrachloroethene	127-18-4	1	10	10	1200

Table 6-A

Target Analyte List for Volatile Organic Compounds by GC/MS

(Based on NYSDEC ASP Requirement for Superfund CLP Program)

Volatile Organic Compound	CAS #	Quantitation Limits – CLP Low Concentration TCL ug/L	Quantitation Limits – CLP TCL		
			Water ug/L	Low Soil ug/kg	Med Soil ug/kg
2-Hexanone	591-78-6	5	10	10	1200
Dibromochloromethane	124-48-1	1	10	10	1200
1,2-Dibromoethane	106-93-4	1	10	10	1200
Chlorobenzene	108-90-7	1	10	10	1200
Ethyl Benzene	100-41-4	1	10	10	1200
Total Xylenes	1330-20-7	--	10	10	1200
Styrene	100-42-5	1	10	10	1200
Bromoform	75-25-2	1	10	10	1200
Isopropylbenzene	98-82-8	--	10	10	1200
1,1,2,2-Tetrachloroethane	79-34-5	1	10	10	1200
1,3-Dichlorobenzene	541-73-1	1	10	10	1200
1,4-Dichlorobenzene	106-46-7	1	10	10	1200
1,2-Dichlorobenzene	95-50-1	1	10	10	1200
1,2-Dibromo-3-chloropropane	96-12-8	1	10	10	1200
1,2,4-Trichlorobenzene	120-82-1	--	10	10	1200
Bromochloromethane	74-97-5	1	--	--	--
o/p-xylene	95-47-6/106-42-3	1	--	--	--
m-xylene	108-38-3	1	--	--	--
Vinyl acetate	108-05-4	1	--	--	--

* Quantitation Limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, as required by the protocol, will be higher.

-- Not on the CLP Low Concentration Organics TCL or CLP TCL.

Table 6-B

Target Analyte List for Semivolatile Organic Compounds by GC/MS

(Based on NYSDEC ASP Requirement for Superfund CLP Program)

Semivolatile Compound	CAS #	Quantitation Limits – CLP Low Concentration TCL ug/L	Quantitation Limits – CLP TCL		
			Water ug/L	Low Soil ug/kg	Med Soil ug/kg
Phenol	108-95-2	5	10	330	10000
bis(2-Chloroethyl) ether	111-44-4	5	10	330	10000
2-Chlorophenol	95-57-8	5	10	330	10000
1,3-Dichlorobenzene	541-73-1	--	10	330	10000
1,4-Dichlorobenzene	106-46-7	--	10	330	10000
1,2-Dichlorobenzene	95-50-1	--	10	330	10000
2-Methylphenol	95-48-7	5	10	330	10000
2,2'-oxybis(1-Chloro-propane)	108-60-1	5	10	330	10000
4-Methylphenol	106-44-5	5	10	330	10000
N-Nitroso-di-n-propylamine	621-64-7	5	10	330	10000
Hexachloroethane	67-72-1	5	10	330	10000
. Nitrobenzene	98-95-3	5	10	330	10000
Isophorone	78-59-1	5	10	330	10000
2-Nitrophenol	88-75-5	5	10	330	10000
2,4-Dimethylphenol	105-67-9	5	10	330	10000
bis(2-Chloroethoxy)methane	111-91-1	5	10	330	10000
2,4-Dichlorophenol	120-83-2	5	10	330	10000
1,2,4-Trichlorobenzene	120-82-1	5	10	330	10000
Naphthalene	91-20-3	5	10	330	10000
4-Chloroaniline	106-47-8	5	10	330	10000
Hexachlorobutadiene	87-68-3	5	10	330	10000
4-Chloro-3-methylphenol	59-50-7	5	10	330	10000
2-Methylnaphthalene	91-57-6	5	10	330	10000
Hexachlorocyclopentadiene	77-47-4	5	10	330	10000
2,4,6-Trichlorophenol	88-06-2	5	10	330	10000
2,4,5-Trichlorophenol	95-95-4	20	25	800	25000
2-Chloronaphthalene	91-58-7	5	10	330	10000
2-Nitroaniline	88-74-4	20	25	800	25000
Dimethyl phthalate	131-11-3	5	10	330	10000
Acenaphthylene	208-96-8	5	10	330	10000
2,6-Dinitrotoluene	606-20-2	5	10	330	10000
3-Nitroaniline	99-09-2	20	25	800	25000
Acenaphthene	83-32-9	5	10	330	10000
2,4-Dinitrophenol	51-28-5	20	25	800	25000

Table 6-B

Target Analyte List for Semivolatile Organic Compounds by GC/MS

(Based on NYSDEC ASP Requirement for Superfund CLP Program)

Semivolatile Compound	CAS #	Quantitation Limits – CLP Low Concentration TCL ug/L	Quantitation Limits – CLP TCL		
			Water ug/L	Low Soil ug/kg	Med Soil ug/kg
4-Nitrophenol	100-02-7	20	25	800	25000
Dibenzofuran	132-64-9	5	10	330	10000
2,4-Dinitrotoluene	121-14-2	5	10	330	10000
Diethylphthalate	84-66-2	5	10	330	10000
4-Chlorophenyl phenyl ether	7005-72-3	5	10	330	10000
Fluorene	86-73-7	5	10	330	10000
4-Nitroaniline	100-01-6	20	25	800	25000
4,6-Dinitro-2-methylphenol	534-52-1	20	25	800	25000
N-nitrosodiphenylamine	86-30-6	5	10	330	10000
4-Bromophenyl phenyl ether	101-55-3	5	10	330	10000
Hexachlorobenzene	118-74-1	5	10	330	10000
Pentachlorophenol	87-86-5	20	25	800	25000
Phenanthrene	85-01-8	5	10	330	10000
Anthracene	120-12-7	5	10	330	10000
Carbazole	86-74-8	--	10	330	10000
Di-n-butyl phthalate	84-74-2	5	10	330	10000
Fluoranthene	206-44-0	5	10	330	10000
Pyrene	129-00-0	5	10	330	10000
Butyl benzyl phthalate	85-68-7	5	10	330	10000
3,3'-Dichlorobenzidine	91-94-1	5	10	330	10000
Benz[a]anthracene	56-55-3	5	10	330	10000
Chrysene	218-01-9	5	10	330	10000
bis(2-Ethylhexyl)phthalate	117-81-7	5	10	330	10000
Di-n-octyl phthalate	117-84-0	5	10	330	10000
Benzo[b]fluoranthene	205-99-2	5	10	330	10000
Benzo[k]fluoranthene	207-08-9	5	10	330	10000
Benzo[a]pyrene	50-32-8	5	10	330	10000
Indeno(1,2,3-cd]pyrene	193-39-5	5	10	330	10000
Dibenz[a,h]anthracene	53-70-3	5	10	330	10000
Benzo[g,h,i]perylene	191-24-2	5	10	330	10000

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the Laboratory for soil/sediment, calculated on dry weight basis as required by the Protocol, will be higher.
-- Not on the CLP Low Concentration Organics TCL.

Table 6-C

Target Analyte List for Explosives by HPLC
(based on SW-846 Method 8330)

Explosive Compound	CAS #	Comments
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	
1,3,5-Trinitrobenzene	99-35-4	
1,3-Dinitrobenzene	99-65-0	
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	479-45-8	
Nitrobenzene	98-95-3	
2,4,6-Trinitrotoluene (TNT)	118-96-7	
4-Amino-2,6-dinitrotoluene	19406-51-0	
2-Amino-4,6-dinitrotoluene	35572-78-2	
2,4-Dinitrotoluene	121-14-2	
2,6-Dinitrotoluene	606-20-2	
2-Nitrotoluene	88-72-2	
3-Nitrotoluene	99-08-1	
4-Nitrotoluene	99-99-0	
Nitroglycerin	55-63-0	Requires SW8332 or modification to SW8330; modification must be identified in SOP
Pentaerythritol Tetranitrate	78-11-5	Requires modification to SW8330; modification must be identified in SOP

Table 6-D

Target Analyte List for Organochlorine Pesticides by GC/ECD

(Based on NYSDEC ASP Requirement for Superfund CLP Program)

Organochlorine Pesticide Compound	CAS #	Quantitation Limits – CLP Low Concentration TCL ug/L	Quantitation Limits – CLP TCL	
			Water ug/L	Soil ug/kg
alpha-BHC	319-84-6	0.01	0.05	1.7
beta-BHC	319-85-7	0.01	0.05	1.7
delta-BHC	319-86-8	0.01	0.05	1.7
gamma-BHC (Lindane)	58-89-9	0.01	0.05	1.7
Heptachlor	76-44-8	0.01	0.05	1.7
Aldrin	309-00-2	0.01	0.05	1.7
Heptachlor epoxide	1024-57-3	0.01	0.05	1.7
Endosulfan I	959-98-8	0.01	0.05	1.7
Dieldrin	60-57-1	0.02	0.10	3.3
4,4'-DDE	72-55-9	0.02	0.10	3.3
Endrin	72-20-8	0.02	0.10	3.3
Endosulfan II	33213-65-9	0.02	0.10	3.3
4,4'-DDD	72-54-8	0.02	0.10	3.3
Endosulfan sulfate	1031-07-8	0.02	0.10	3.3
4,4'-DDT	50-29-3	0.02	0.10	3.3
Methoxychlor	72-43-5	0.10	0.50	17.0
Endrin ketone	53494-70-5	0.02	0.10	3.3
Endrin aldehyde	7421-36-3	0.02	0.10	3.3
alpha-Chlordane	5103-71-9	0.01	0.05	1.7
gamma-Chlordane	5103-74-2	0.01	0.05	1.7
Toxaphene	8001-35-2	1.0	5.0	170.0

Note: Quantitation Limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the Laboratory for soil/sediment, calculate on dry weight basis, as required by the Protocol, will be higher.

Table 6-E

Target Analyte List for Polychlorinated Biphenyls by GC/ECD

(Based on NYSDEC ASP Requirement for Superfund CLP Program)

PCB Compound	CAS #	Quantitation Limits – CLP Low Concentration TCL ug/L	Quantitation Limits – CLP TCL	
			Water ug/L	Soil ug/kg
Aroclor 1016	12674-11-2	0.2	1	33
Aroclor 1221	11104-28-2	0.2	2	67
Aroclor 1232	11141-16-5	0.4	1	33
Aroclor 1242	53469-21-9	0.2	1	33
Aroclor 1248	12672-29-6	0.2	1	33
Aroclor 1254	11097-69-1	0.2	1	33
Aroclor 1260	11096-82-5	0.2	1	33

Note: Quantitation Limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the Laboratory for soil/sediment, calculate on dry weight basis, as required by the Protocol, will be higher

Table 6-F

Target Analyte List for Inorganics
(Based on NYSDEC ASP Requirement for Superfund CLP Program[1])

Metal	CAS #	Contract Required Quantitation Level (µg/L)	Comments
Aluminum	7429-90-5	200	6010B/6020A/7020
Antimony	7440-36-0	60	6010B//6020A/7041
Arsenic	7440-38-2	10	6010B/6020A/7060A/7061 A
Barium	7440-39-3	200	6010B/6020A/7081
Beryllium	7440-41-7	5	6010B//6020A//7091
Cadmium	7440-43-9	5	6010B/6020A/7131A
Calcium	7440-70-2	5000	6010B/6020A
Chromium	7440-47-3	10	6010B/6020A/7191
Cobalt	7440-48-4	50	6010B/6020A/7201
Copper	7440-50-8	25	6010B/6020A/7211
Iron	7439-89-6	100	6010B/6020A/7381
Lead	7439-92-1	3	6010B/6020A/7421
Magnesium	7439-95-4	5000	6010B/6020A
Manganese	7439-96-5	15	6010B/6020A/7461
Mercury	7439-97-6	0.2	7470/7471/7472/6020A
Nickel	7440-02-0	40	6010B/6020A/7521
Potassium	7440-09-7	5000	6010B/6020A
Selenium	7782-49-2	5	6010B//6020A/7740
Silver	7440-22-4	10	6010B/6020A/7761
Sodium	7440-23-5	5000	6010B/6020A
Strontium	7440-24-6		6010B
Thallium	7440-28-0	10	6010B/6020A/7841
Vanadium	7440-62-2	50	6010B/7911/6020A
Zinc	7440-66-6	20	6010B/6020A/7951
Cyanide	7440-67-7	10	9010/9012A/9013/9014

(1) Site-specific work plans must specify which method is intended.

Table 6-G
Target Analyte List for RCRA TCLP Test
(Based on NYSDEC ASP Requirement for RCRA TCLP Program[)])

Analyte	CAS #	Contract Required Quantitation Level (µg/L)
Metals		
Arsenic	7440-38-2	1000
Barium	7440-39-3	10000
Cadmium	7440-43-9	100
Chromium (total)	7440-47-3	1000
Lead	7439-92-1	1000
Mercury	7439-97-6	50
Selenium	7782-49-2	100
Silver	7440-22-4	1000
Volatiles		
Benzene	71-43-2	10
2-Butanone (Methylethylketone)	78-93-3	10
Carbon tetrachloride	56-23-5	10
Chlorobenzene	108-90-7	10
Chloroform	67-66-3	10
1,2-Dichloroethane	107-06-2	10
1,1-Dichloroethylene	75-35-4	10
Tetrachloroethylene	127-18-4	10
Trichloroethylene	79-01-6	10
Vinyl chloride	75-01-4	10
Semivolatiles		
1,4-Dichlorobenzene	106-46-7	10
2,4-Dinitrotoluene	121-14-2	10
Hexachlorobenzene	118-74-1	10
Hexachlorobutadiene	87-68-3	10
Hexachloroethane	67-72-1	100
2-Methylphenol (o-Cresol)	95-48-7	10
3-Methylphenol (m-Cresol)	108-39-4	10
4-Methylphenol (p-Cresol)	106-44-5	10
Nitrobenzene	98-95-3	10
Pentachlorophenol	87-86-5	5
Pyridine	110-86-1	100
2,4,5-Trichlorophenol	95-95-4	10
2,4,6-Trichlorophenol	88-06-2	10
Pesticides		
Gamma-BHC (Lindane)	58-89-9	10
Chlordane	57-74-9	10
Endrin	72-20-8	0.5
Heptachlor	76-44-8	0.5
Heptachlor Epoxide	1024-57-3	0.5
Methoxychlor	72-43-5	100
Toxaphene	8001-35-2	10
Herbicides		
2,4-Dichlorophenoxyacetic acid (2,4-D)	94-75-7	100
2,4,5-Trichlorophenoxypropionic acid (2,4,5-TP; Silvex)	93-76-5	10

Table 7-A
Quality Control Requirements for Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography
(Methods 7580, 8081A, 8082, and 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method	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	Not applicable (NA)	This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
Method detection limit (MDL) study	At initial set-up and subsequently once per 12 month period; otherwise quarterly MDL verification checks shall be performed	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument's noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study	NA	Samples cannot be analyzed without a valid MDL.
Retention time window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	Width is ± 3 times standard deviation for each analyte retention time from 72-hour study.	NA	NA	

Table 7-A
Quality Control Requirements for Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography
(Methods 7580, 8081A, 8082, and 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Breakdown check (Endrin/ DDT Method 8081A only)	Daily prior to analysis of samples	Degradation < 20% for either endrin or DDT; Degradation <30% for sum of endrin and DDT.	Correct problem, then repeat breakdown check.	If 4,4'-DDT breakdown>20%: i. Qualify all positive DDT results with 'J". If DDT was not detected, but DDD/DDE are positive, qualify the QL for DDT as "R". ii. Qualify positive results for DDD and DDE as "NJ". b. If endrin breakdown>20%: i. Qualify all positive results for endrin with "J". If endrin was not detected, but endrin aldehyde and endrin ketone are positive, then qualify the QL for endrin as "R". ii. Qualify positive results for endrin ketone and endrin aldehyde as "NJ".	No samples shall be run until degradation for individual compound DDT and endrin < 20% and degradation for combined DDT and endrin <30%.

Table 7-A
Quality Control Requirements for Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography
(Methods 7580, 8081A, 8082, and 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Minimum three-point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis. For HPLC, a five point initial calibration is required.	1) All single component analytes **except** alpha and delta BHC must be equal or less than the maximum %RSD of 20%. 2) Alpha and delta BHC must be equal or less than the maximum %RSD of 25%. 3) Surrogates must be equal or less than the maximum %RSD of 30%. 4) The SOW allows up to **2** of all single component analytes except the surrogates to fail contractual requirements for %RSD. **The failing analytes must have a %RSD < 30%.**	Correct problem, then repeat initial calibration.	If technical criteria were not met, qualify all associated positive results generated during the entire analytical sequence "J" and all non-detects "UJ". When %RSD > 90%, flag all non-detect results for that analyte "R" (unusable), and positive results as "J" estimated**.**	Problem must be corrected. No samples may be run until ICAL has passed. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of another Aroclor (e.g., 1268, 1262). In addition, a mid-level or lower standard for each of the remaining Aroclors is analyzed for pattern recognition and response factor.

Table 7-A
Quality Control Requirements for Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography
(Methods 7580, 8081A, 8082, and 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 25% of expected value (initial source)	Correct problem and verify second source standard. If that fails then repeat initial calibration.	If the %D is outside the ±25.0% range for any compound(s), qualify associated positive results for that compound "J" and non-detects "UJ". The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyte(s) in question. If the %D is > 90%, flag all nondetects for that analyte "R" (unusable).	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL	The center of the retention time window shall be set at midpoint of initial calibration curve.	NA	NA	
Retention time window verification for each analyte and surrogate	Each calibration verification standard	All analytes and surrogates within established windows	Correct problem, then reprocess all samples analyzed since the last acceptable retention time check. Or, perform a new ICAL and reset retention time windows.	Flagging criteria is not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window.	No samples shall be run without correctly set retention time windows.

Table 7-A
Quality Control Requirements for Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography
(Methods 7580, 8081A, 8082, and 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Calibration verification (initial [ICV] and continuing [CCV])	An instrument blank and the PEM must bracket one end of a 12-hour period during which sample data are collected, and a second instrument blank and the midpoint concentration of Individual Standard Mixtures A and B must bracket the other end of the 12-hour period.	All analytes within ± 25% of expected value (%D).	Correct problem, rerun calibration verification and reanalyze all samples since last successful calibration verification.	If the %D is outside the ±25.0% range for any compound(s), qualify associated positive results for that compound "J" and non-detects "UJ". The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyte(s) in question. If the %D is > 90%, flag all nondetects for that analyte "R" (unusable).	If an individual analyte is > 25%, no samples may be analyzed until the problem has been corrected.
Method blank	A method blank must be extracted each time 20 or less field samples (excluding matrix spikes/matrix spike duplicates and PE samples) are extracted. In addition, a method blank shall be analyzed on each GC/EC system used to analyze associated samples	No analytes detected ≥ CRQL and surrogate recoveries within 30%~150%.	Correct problem, if required, reprep then reanalyze method blank and all samples processed with the contaminated blank.	Flag sample result with a U if sample > CRQL but < or =5 x blank level; Report CRQL and qualify U if sample<CRQL and < or = 5 x blank level; no action if sample >CRQL and > 5 x CRQL.	

Table 7-A
Quality Control Requirements for Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography
(Methods 7580, 8081A, 8082, and 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Instrument blank	First analysis in a 12-hr analysis sequence	No analytes detected ≥ CRQL and surrogate recoveries within 30%~150%.	Correct problem, then reanalyze instrument blank and all samples associated with the contaminated blank.		
Laboratory control sample (LCS) containing all analytes required to be reported by the project or contract	One LCS for every group of samples in the Sample Delivery Group.	QC acceptance criteria specified in CLP and those specified in Table 9.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated preparatory batch, if sufficient sample material is available	If corrective action fails, or sufficient sample volume is not available for reprep, apply professional judgment to determine necessary quailifier for specific analyte(s) in all samples in the associated preparatory batch.	LCS technical acceptance criteria MUST be met before data are reported. LCS contamination from laboratory sources or any LCS analyzed not meeting the technical acceptance criteria will require re-extraction and re-analysis of the LCS at no additional cost.

Table 7-A
Quality Control Requirements for Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography
(Methods 7580, 8081A, 8082, and 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Matrix spike (MS)	A matrix spike must be performed for the following, whichever is most frequent: 1) Each SDG, 2) Each group of 20 field samples. 3) Each group of field samples of a similar concentration level.	For matrix evaluation, use QC acceptance criteria specified by CLP and those specified in Table 9.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	No action is taken based upon MS/MSD data alone. However, using informed professional judgment, the MS/MSD results may be used in conjunction with other QC criteria to determine the need for qualification of the data. The validator using **professional judgment** has several options: 1) Do nothing 2) Qualify only the affected analyte in the unspiked sample 3) Qualify all of the analytes in the unspiked sample 4) Qualify only the affected analyte in all samples. This must have supporting details to document this action 5) Qualify only the affected analyte or all of the analytes if recovery is < 10%	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table 7-A
Quality Control Requirements for Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography
(Methods 7580, 8081A, 8082, and 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	A matrix spike duplicate or sample duplicate must be performed for the following, whichever is most frequent: 1) Each SDG, 2) Each group of 20 field samples, 3) Each group of field samples of a similar concentration level. For 8330, one laboratory duplicate should be submitted for each extraction batch.	RPD (between MS and MSD or sample and sample duplicate) meets criteria specified in Table 9.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	Professional judgment, see above.	The data shall be evaluated to determine the source of difference.

Table 7-A
Quality Control Requirements for Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography
(Methods 7580, 8081A, 8082, and 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Surrogate spike (analytes identified in Table 8)	All field and QC samples	QC acceptance criteria for surrogate specified in Table 8	For QC and field samples, reanalyze sample. If surrogate recovery is still out, identify the cause of the problem. If possible, 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.	Qualify data in accordance with Region II SOP. In brief, 1) If surrogate on both columns is below limit but >10%, check chromatograms for interference and qualify affected analytes 2) If surrogate on both columns is below limit but > 10%, J non-detects and positive hits. 3) If recoveries for both surrogates on both columns are below limit but >10%, J positive results and UJ non-detects. 4) If recoveries are above limit for both surrogates on both columns, J positive values. 5) If both surrogates on one column are below limit but > 10%, use the data from the other column, providing both surrogates on that column are within limits. 6) If recovery is <10% for either surrogate on any column, and no chromatographic or matrix interference is visible, J positive results and R non-detects.	Alternative surrogates are recommended when there is obvious chromatographic interference.

Table 7-A
Quality Control Requirements for Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography
(Methods 7580, 8081A, 8082, and 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Confirmation of positive results (second column or second detector)	All analytes detected above the MDL require confirmation on a second column or second detector.	Results between two columns %D ≤ 25%. (when higher value is compared to lower value)	NA	Qualify data in accordance with Region II SOP. In brief, %D=0 - 25%, no action %D=26 - 70%, "J" %D=71 - 100%, "JN" %D=100-200% (No Interference) "R" %D=100 - 200% (Interference detected), "JN" %D> 50% (Pesticide value is < CRQL), "U" %D> 200% "R"	Report the lower of two results unless QA/QC issues with the column.
Results reported between MDL and RL	NA	NA	NA	Apply J to all results below CRQL. Hits well below the CRQLs (less than 1/2 the CRQL value) may be column/background noise and using discretion may not be reported.	

Table 7-B
Quality Control Requirements for Organic Analysis by Gas Chromatography/Mass Spectroscopy (Methods 8260B, 8270C, 524.2) and GC/FID (Method 8015B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method	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 ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument's noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study	NA	Samples cannot be analyzed without a valid MDL.

Table 7-B
Quality Control Requirements for Organic Analysis by Gas Chromatography/Mass Spectroscopy (Methods 8260B, 8270C, 524.2) and GC/FID (Method 8015B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Tuning (MS methods only)	Prior to calibration and every 12 hours during sample analysis	Refer to Table 1 in Exhibit D of CLP OLC03.2 (December 2000) and OLM04.3 (March 2003) for specific requirements.	Retune instrument and verify. Rerun affected samples.	Flagging criteria is not appropriate	Problem must be corrected. No samples may be accepted without a valid tune.
Minimum five-point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis, whenever corrective action is taken which may affect initial calibration, and whenever calibration acceptance criteria have not been met.	Refer to CLP OLC03.2 (December 2000) and OLM04.3 (March 2003) for specific requirements. VOC: Table 5 in Exhibit D, OLM04.3; Table D-2 in Exhibit D, OLC03.2 SVOC: Table 5 in Exhibit D, OLM04.3; Table D-4 in Exhibit D, OLC03.2 Up to two compounds may fail the criteria and still meet the minimum RRF and %RSD requirements. However, these compounds must have a minimum RRF greater than or equal to 0.010, and the %RSD must be less than or equal to 40.0 percent.	Correct problem then repeat initial calibration.	Qualify in accordance with Region II SOP. In brief: If %RSD is > 30.0%, qualify associated positive results for that analyte "J". When %RSD is > 90%, flag all non-detects for that analyte "R" and positive hits "J" **.** If the average RRF is < 0.05, qualify associated non-detects with an "R" and flag associated positive data as estimated "J".	Problem must be corrected. No samples may be run until ICAL has passed.
Retention time window position establishment for each analyte and surrogate	Once per ICAL	Position shall be set using the midpoint standard of the initial calibration curve.	NA	NA	
Evaluation of	Each	All analytes and surrogates within established	Correct problem, then	Use professional	

Table 7-B
Quality Control Requirements for Organic Analysis by Gas Chromatography/Mass Spectroscopy (Methods 8260B, 8270C, 524.2) and GC/FID (Method 8015B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
relative retention times (RRT)	calibration verification standard	windows	reprocess all samples analyzed since the last acceptable retention time check. Or, perform a new ICAL and reset retention time windows.	judgment. If it is determined that incorrect identifications were made, all such data should be rejected "R", flagged "N" or changed to not detected "U" at the calculated detection limit.	
Calibration verification (CV)	Daily, before sample analysis, after injection of instrument performance compound and every 12 hours of analysis time	Refer to CLP OLC03.2 (December 2000) and OLM04.3 (March 2003) for specific requirements. VOC: Table 5 in Exhibit D, OLM04.3; Table D-2 in Exhibit D, OLC03.2 SVOC: Table 5 in Exhibit D, OLM04.3; Table D-4 in Exhibit D, OLC03.2 Up to two compounds may fail the criteria and still meet the minimum RRF and %RSD requirements. However, these compounds must have a minimum RRF greater than or equal to 0.010, and the %RSD must be less than or equal to 40.0 percent.	Correct problem, rerun CV. If that fails, then repeat initial calibration and reanalyze all affected samples.	Qualify both positive results and non-detects for the outlier compound(s) as estimated if %D>25%. When %D is > 90%, qualify all non-detects for that analyte unusable (R) and positive results estimated (J) **.** If any RRF is < 0.05, qualify the associated non-detects as unusable "R" and the associated positive values "J"..	

Table 7-B
Quality Control Requirements for Organic Analysis by Gas Chromatography/Mass Spectroscopy (Methods 8260B, 8270C, 524.2) and GC/FID (Method 8015B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Internal standards	With every continuing calibration, and with every project sample	Internal standard areas of every sample and blank should be within the upper and lower limits (-50% to +100%) for each continuing calibration; the retention times of the internal standards should be within 30 seconds of the associated calibration standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory. See corrective action for CV.	Qualify in accordance with Region II SOP. In brief, if the IS area count is outside limit, "J" positive results quantitated with this IS. Do not qualify nondetects when associated IS area counts are > 100%. If IS area is < 50%, qualify all associated analytes "J". If the area counts are < 25% of the area in the 12 hour IS, or if performance exhibits a major abrupt dropoff, flag associated nondetects "R" and positive hits "J".	Sample results are not acceptable without a valid CV-IS.

Table 7-B
Quality Control Requirements for Organic Analysis by Gas Chromatography/Mass Spectroscopy (Methods 8260B, 8270C, 524.2) and GC/FID (Method 8015B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Method blank	SVOC: One per preparatory batch (or 20 field samples, whichever is more frequent), VOC: analyzed every 12-hr time period on each GC/MS system, before any samples, and for each matrix.	The concentration of each target compound found in the storage and method blanks must be less than its CRQL, except for methylene chloride and cyclohexane which must be less than 10 times their respective CRQLs, acetone and 2-butanone, which must be less than two times their respective CRQLs, and phthalate esters, which must be less than 5 times CRQLs.	Correct problem. All samples processed within the 12-hour time period with a method blank or instrument blank that does not meet the blank technical acceptance criteria will require reanalysis at no additional cost.	For common analytes, flag sample result with a U if sample >CRQL but ≤10xblank level; Report CRQL and qualify U if sample<CRQL and ≤10xblank level; no action if sample >CRQL and >10xCRQL. For other analytes, flag sample result with a U if sample > CRQL but ≤5xblank level; Report CRQL and qualify U if sample<CRQL and ≤5xblank level; no action if sample >CRQL and >5xCRQL.	The source of the contamination must be investigated and appropriate corrective measures MUST be taken and documented before further sample analysis proceeds.

Table 7-B
Quality Control Requirements for Organic Analysis by Gas Chromatography/Mass Spectroscopy (Methods 8260B, 8270C, 524.2) and GC/FID (Method 8015B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
LCS containing all analytes required to be reported by the project or CLP	One LCS per preparatory batch	QC acceptance criteria specified by the laboratory or 70~130% when laboratory advisory limits are not available.	Correct problem, then reprep and reanalyze the LCS and all associated samples for failed analytes, if sufficient sample material is available.	When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.	When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

Table 7-B
Quality Control Requirements for Organic Analysis by Gas Chromatography/Mass Spectroscopy (Methods 8260B, 8270C, 524.2) and GC/FID (Method 8015B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
MS	A matrix spike must be performed for the following, whichever is most frequent: 1) Each SDG, 2) Each group of 20 field samples, 3) Each group of field samples of a similar concentration level.	For matrix evaluation, use QC acceptance criteria specified by CLP and those specified in Table 9.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	No action is taken based upon MS/MSD data alone. However, using informed professional judgment, the MS/MSD results may be used in conjunction with other QC criteria to determine the need for qualification of the data. The validator using **professional judgment** has several options: 1) Do nothing 2) Qualify only the affected analyte in the unspiked sample 3) Qualify all of the analytes in the unspiked sample 4) Qualify only the affected analyte in all samples. This must have supporting details to document this action 5) Qualify only the affected analyte or all of the analytes if recovery is < 10%	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

Table 7-B
Quality Control Requirements for Organic Analysis by Gas Chromatography/Mass Spectroscopy (Methods 8260B, 8270C, 524.2) and GC/FID (Method 8015B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
MSD or sample duplicate	A matrix spike duplicate or sample duplicate must be performed for the following, whichever is most frequent: 1) Each SDG, 2) Each group of 20 field samples, 3) Each group of field samples of a similar concentration level.	RPD (between MS and MSD or sample and sample duplicate) meets criteria specified in Table 9.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	Professional judgment, see above.	The data shall be evaluated to determine the source of difference.

Table 7-B
Quality Control Requirements for Organic Analysis by Gas Chromatography/Mass Spectroscopy (Methods 8260B, 8270C, 524.2) and GC/FID (Method 8015B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Surrogate spike (analytes identified in Table 8)	All field and QC samples	QC acceptance criteria for in Table 8.	For VOC, if any surrogate recovery fails, correct problem, then all samples with failing surrogates must be reanalyzed. For SVOC, if two or more surrogate within the same fraction fail, correct problem, then all samples with failing surrogates in the associated preparatory batch must be reprepped and /or reanalyzed.	For VOC, if recoveries are ≥10%, but surrogate recoveries outside limits, all positive results are qualified as "J"; non-detects are flagged as "UJ" where recovery is less than lower limit. If any surrogate recovery is <10%, flag all positive results as J, and all non-detects as R. For SVOC, if two BN or acid surrogate recoveries exceed limits but are ≥10%, for the affected fraction only, qualify positive results as J and flag all non-detects as UJ when recoveries are less than lower limit. If any surrogate recovery is < 10%, qualify associated positive results as J and non-detects as R.	
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL.	

Table 7-C
Quality Control Requirements for Inorganic Analysis by Inductively Coupled Plasma (ICP) and Atomic Absorption Spectroscopy (AA) (Methods 6010B And 7000A Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method	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 analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study	NA	Samples cannot be analyzed without a valid MDL.
Instrument detection limit (IDL) study (ICP only)	Every 3 months	Detection limits established shall be ≤ CRDL.	NA	NA	Samples cannot be analyzed without a valid IDL.
Linear range or high-level calibration check standard (ICP only)	The upper limit of the linear range should be established prior to the start of contract analyses and at least quarterly thereafter.	NA	NA	NA	No samples may be analyzed without a valid upper limit of linear range established.

Table 7-C
Quality Control Requirements for Inorganic Analysis by Inductively Coupled Plasma (ICP) and Atomic Absorption Spectroscopy (AA) (Methods 6010B And 7000A Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Initial calibration for all analytes (ICAL)	Daily initial calibration prior to sample analysis (ICP: minimum one high standard and a blank; GFAA: minimum three standards and a blank; CVAA: minimum 4 standards and a blank)	ICP: No acceptance criteria unless more than one standard is used, in which case $r \geq 0.995$. GFAA: $r \geq 0.995$ CVAA: $r \geq 0.995$	Correct problem and repeat initial calibration.	Flag as J all associated results.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each initial calibration, prior to sample analysis	All analyte(s) within ± 10% of expected value.	Correct problem and verify second source standard. If that fails, then repeat initial calibration.	Flag as J all positive data with %R between 75-89% (65-79% for Hg; 70-84% for CN) or 111-125% (121-135% for Hg; 116-130% for CN) recovery. Qualify results <IDL as UJ if ICV %R is 75-89% (CN, 70-84%; HG, 65-79%). Reject data if recovery of the ICV is outside the range 75-125% (CN, 70-130%; Hg, 65-135%). Qualify five samples on either side of verification standard out of control limits.	Problem must be corrected. No samples may be run until calibration has been verified as acceptable.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analyte(s) within ± 10% of expected value	Correct problem, rerun calibration verification. If that fails, repeat initial calibration and reanalyze all samples since last successful calibration.	Save as above.	Problem must be corrected. Results may not be reported without a valid CCV.

Table 7-C
Quality Control Requirements for Inorganic Analysis by Inductively Coupled Plasma (ICP) and Atomic Absorption Spectroscopy (AA) (Methods 6010B And 7000A Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Low level calibration check standard (ICP only)	Daily after ICV/ICB and immediately preceding the Interference Check Sample (ICS) analyses; in addition, at the end of each sample analysis run and at a frequency of not less than once per 20 analytical samples[1] per analysis run, followed by CCV/CCB.	The percent recovery of the CRI should fall within 70-130% (50-150% for antimony, lead, and thallium)	Reanalyze CRI, if still not within the limits, correct problem, recalibrate instrument, then reanalyze.	If the recovery of the standard is between 50-69%, flag all positive sample results as "J" and all non-detect results as "UJ"; If the recovery is between 131-150%, flag positive sample results as "J"; If the recovery is less than 50%, flag all data as "R"; If the recovery is greater than 150%, flag all positive sample results as "R".	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. If a multipoint calibration is performed and the low point of the calibration is at or below the reporting limit, no low level calibration check is necessary.

Table 7-C
Quality Control Requirements for Inorganic Analysis by Inductively Coupled Plasma (ICP) and Atomic Absorption Spectroscopy (AA) (Methods 6010B And 7000A Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Preparation blank	One per preparatory batch, or one per SDG, whichever is more frequent.	No analytes detected ≥ CRDL. For common laboratory contaminants, no analytes detected ≥ RL.	If preparation blank>CRDL, all associated samples with concentrations less than 10 times the blank and above the CRQL shall be redigested and re-analyzed with appropriate new Quality Control (QC) for that analyte. If preparation blank < -CRDL, all samples reported below 10 x CRDL associated with the blank, shall be redigested and re-analyzed with appropriate new QC.	Flagging criteria is not appropriate.	
Calibration blank	Immediately after every ICV and CCV, at a frequency of 10% or every 2 hours during the run, whichever is more frequent.	No analytes detected ≥ CRDL or 2xIDL if IDL > CRDL.	Correct problem, then reanalyze calibration blank and all associated samples.	Flag as (J) positive sample results when raw sample value is less than or equal to calibration blank value analyzed between calibration blank with value over CRDL (or 2xIDL) and nearest good calibration blank. Flag five samples on either side of the calibration blank outside limits.	
Interference check solutions (ICP only)	At the beginning (after ICV) and end of an analytical run,	Within ±20% of true value	Terminate analysis, locate and correct problem, recalibrate	If ICS recovery is between 121-150%, flag associated positive sample results "J"; if ICS	

Table 7-C
Quality Control Requirements for Inorganic Analysis by Inductively Coupled Plasma (ICP) and Atomic Absorption Spectroscopy (AA) (Methods 6010B And 7000A Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
	and at a frequency of not less than once per 20 analytical samples per analysis run.		instrument, reanalyze ICS, reanalyze all affected samples.	recovery falls within 50-79%, flag associated positive sample results as "J" and associated non-detect sample results as "UJ"; If ICS recovery is <50%, flag all associated results as "R"; if ICS recovery is > 150%, flag all positive results as "R".	
LCS containing all analytes required to be reported by the project or contract	One LCS per preparatory batch, or per SDG, whichever is more frequent for each matrix (aqueous and solid).	Within ±20% of true value for aqueous LCS. Within the USEPA specified limits for solid LCS provided by USEPA.	If the % recovery for aqueous LCS falls outside 80 - 120% (exception: Ag and Sb), or if the results for the solid LCS fall outside the EPA limits, the analyses must be terminated, the problem corrected, and the previous samples associated with the LCS redigested and reanalyzed..	If aqueous LCS recovery is < 50%, reject all data; flag associated data as J if LCS recovery is between 50% and 79%; flag all positive results as J if recovery is between 121% and 150%; reject all positive results if recovery is greater than 150%. If solid LCS recovery is higher than limits, qualify all associated positive data as J. If solid LCS recovery is lower than limits, qualify all associated data as J.	

Table 7-C
Quality Control Requirements for Inorganic Analysis by Inductively Coupled Plasma (ICP) and Atomic Absorption Spectroscopy (AA) (Methods 6010B And 7000A Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Serial dilution test (ICP only)	One sample from each SDG and matrix.	Five-fold dilution must agree within ± 10% of the original measurement if concentration is sufficiently high (i.e., >10IDL).		Flag as J all the associated sample data > 10xIDLs (or > CRDL when 10xIDL < CRDL) for which percent difference is greater than 10% but less than 100%. Reject all the associated sample results equal to or greater than 10xIDLs (or > CRDL when 10xIDL < CRDL) for which %D is greater than or equal to 100%.	Only applicable for samples with concentrations > 10 x IDL.
Post digestion spike (ICP and cyanide)	When MS test fails.	Recovery within 75-125% of expected results	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	No action is taken based upon PDS data alone. However, using informed professional judgment, the PDS results may be used in conjunction with other QC criteria to determine the need for qualification of the data.	
Recovery test (GFAA only)	When dilution test fails.	Recovery within 85-115% of expected results.	Run samples by method of standard addition (MSA) or see flagging criteria.	Apply J to all sample results (for same matrix) in which MSA was not run when recovery is outside of 85-115% range.	
Method of standard addition (MSA)	When matrix interference is suspected	NA	NA	NA	Document use in the case narrative.

Table 7-C
Quality Control Requirements for Inorganic Analysis by Inductively Coupled Plasma (ICP) and Atomic Absorption Spectroscopy (AA) (Methods 6010B And 7000A Series)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
MS	One MS every 20 project samples, or per SDG, whichever is more frequent per matrix.	Recovery within 75-125% of expected results	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For aqueous, if < 30%, reject all associated results; if between 30-74%, flag all associated results as "J"; if between 126-150%, flag all associated positive results as "J"; if >150%, reject all associated positive sample results. For solid samples, if <10%, reject all associated data; if between 10-74%, flag all associated data as "J"; if between 126-200%, flag all associated positive results as "J"; if >200%, reject all associated positive results.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD or sample duplicate	One every 20 project samples, or per SDG, whichever is more frequent, per matrix	RPD < 20%, or difference <CRDL when both results<5CRDL(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 if acceptance criteria are not met (RPD<50% for aqueous and RPD<100% for soil; difference<CRDL for aqueous and difference<2CRDL for soil when both concentrations <5CRDL).	The data shall be evaluated to determine the source of difference.
Results reported between IDL and CRDL	NA	NA	NA	Apply J to all results between IDL and CRDL.	

Table 7-D
Quality Control Requirements for Trace Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (Method 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel or test method	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 analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and once per 12 months for each digestion approach and instrument used, and after major instrument maintenance/ instrumental condition change.	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study.	NA	Samples cannot be analyzed without a valid MDL.
IDL study	Every 3 months	Detection limits established shall be ≤ CRDL.	NA	NA	Samples cannot be analyzed without a valid IDL.
Tuning (MS methods only)	Prior to initial calibration	Per 6020 (5.8) and CLP ILM05.3	Retune instrument then reanalyze tuning solutions.	Flagging criteria is not appropriate.	No analysis shall be performed without a valid MS tune.

Table 7-D
Quality Control Requirements for Trace Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (Method 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Initial calibration (ICAL) **(minimum one high standard and a blank)**	Daily initial calibration prior to sample analysis, or after changes or corrections to the analytical system.	If more than one calibration standard is used, $r \geq 0.995$	Correct problem, then repeat initial calibration.	Flag as J all associated results.	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 analytes within ± 10% of expected value (initial source)	Correct problem and verify second source standard. If that fails, then repeat initial calibration.	Flag as J all positive data (not flagged with a "U") analyzed between a calibration verification with %R between 75-89% or 111-125% recovery and nearest good calibration standard. Qualify results <IDL as estimated (UJ) if the ICV is 75-89%. Reject data if recovery of the ICV is outside the range 75-125%. Qualify five samples on either side of verification standard out of control limits.	Problem must be corrected. No samples may be run until calibration has been verified.

Table 7-D
Quality Control Requirements for Trace Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (Method 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within ± 10% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration.	Flag as J all positive data (not flagged with a "U") analyzed between a calibration verification with %R between 75-89% or 111-125% recovery and nearest good calibration standard. Qualify results <IDL as estimated (UJ) if the ICV is 75-89%. Reject data if recovery of the CCV is outside the range 75-125%. Qualify five samples on either side of verification standard out of control limits.	Problem must be corrected. Results may not be reported without a valid CCV.
Low-level calibration check standard (CRI)	Daily, after ICV/ICB and immediately preceding the Interference Check Sample (ICS) analyses. In addition, the lab shall analyze the CRI at the end of each sample analysis run and at a frequency of not less than once per 20 analytical samples[1] per analysis run. These subsequent analyses of the CRI shall be immediately followed by CCV/CCB analyses.	Within ± 30% of expected value (within ± 50% for cobalt, manganese, and zinc).	Correct problem, then reanalyze.	Flag as J all sample results within the affected range if the recovery of the standard is between 50-69%; flag only positive data within the affected range if the recovery is between 131-150%; reject all data within the affected range if the recovery is less than 50%; reject only positive data within the affected range if the recovery is greater than 150%. Qualify 50% of the samples on either side of CRI standard outside the control limits.	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.

Table 7-D
Quality Control Requirements for Trace Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (Method 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Linear range or high-level calibration check standard	The upper limit of the linear range should be established prior to the start of contract analyses and at least quarterly thereafter.	NA	NA	NA	No samples may be analyzed without a valid upper limit of linear range established.
Preparation blank	One per preparatory batch, or one per SDG, whichever is more frequent.	No analytes detected ≥ CRDL. For common laboratory contaminants, no analytes detected ≥ RL.	If preparation blank>CRDL, all associated samples with concentrations less than 10 times the blank concentration and above the CRQL shall be redigested and re-analyzed with appropriate new Quality Control (QC) for that analyte. If preparation blank < -CRDL, all samples reported below 10 x CRDL associated with the blank, shall be redigested and re-analyzed with appropriate new QC.	Flagging criteria is not appropriate.	No samples may be analyzed without an acceptable method blank.

Table 7-D
Quality Control Requirements for Trace Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (Method 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
Calibration blank	Immediately after every ICV and CCV, at a frequency of 10% or every 2 hours during the run, whichever is more frequent.	No analytes detected ≥ CRDL or 2IDL if IDL > CRDL.	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples.	Flag as (J) positive sample results when raw sample value is less than or equal to calibration blank value analyzed between calibration blank with value over CRDL (or 2xIDL) and nearest good calibration blank. Flag five samples on either side of the calibration blank outside limits.	
Interference check solutions (ICS-A and ICS-AB)	The interference check solutions shall be analyzed at the beginning (after ICV) and end of an analytical run or twice during an 8-hour working shift, whichever is more frequent.	Within ±20% of true value, or ±3 times the CRQL, whichever is greater.	Terminate analysis, locate and correct problem, recalibrate instrument, reanalyze ICS, reanalyze all affected samples.	If recovery is between 121-150%, flag all positive results "J"; If recovery falls within 50-79%, flag all results as "J"; If recovery is <50%, flag all results "R"; if ICS recovery is > 150%, flag all positive results as "R".	
LCS containing all analytes required to be reported by the project or contract	One LCS per preparatory batch, or per SDG, whichever is more frequent for each matrix (aqueous and solid)..	Within ±20% of true value for aqueous LCS. Within the USEPA specified limits for solid LCS provided by USEPA.	If the % recovery for aqueous LCS falls outside 80 - 120% (exception: Ag and Sb), or if the results for the solid LCS fall outside the EPA limits, the analyses must be terminated, the problem corrected, and the previous samples associated with the LCS redigested and reanalyzed..	If aqueous LCS recovery is < 50%, reject all data; if LCS recovery is between 50% and 79% flag associated data as "J"; if recovery is between 121% and 150% flag all positive results as "J"; if recovery is greater than 150% reject all positive results. If solid LCS recovery is higher than limits, qualify all associated positive data as J. If solid LCS recovery is lower than limits, qualify all associated data as J.	
Serial dilution	One sample from each	Five-fold dilution		Flag as J all the associated	Only applicable for

Table 7-D
Quality Control Requirements for Trace Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (Method 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
test	SDG and matrix.	must agree within ± 10% of the original measurement if concentration is sufficiently high (i.e., >10IDL).		sample data > 10xIDLs (or > CRDL when 10xIDL < CRDL) for which percent difference is greater than 10% but less than 100%. Reject all the associated sample results equal to or greater than 10xIDLs (or > CRDL when 10xIDL < CRDL) for which %D is greater than or equal to 100%.	samples with concentrations > 10 x IDL.
Post digestion spike	When MS test fails.	Recovery within 75-125% of expected results	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	NA.	
MS	One MS every 20 project samples, or per SDG, whichever is more frequent per matrix.	Recovery within 75-125% of expected results	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For aqueous, if < 30%, reject all associated aqueous data; if between 30-74%, flag all associated aqueous data as "J"; if between 126-150%, flag all positive sample results as "J"; if >150%, reject all positive sample results. For solid samples, if <10%, reject all associated data; if between 10-74%, flag all associated data as "J"; if between 126-200%, flag as "J" all positive sample results; if >200%, reject all positive sample results.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD or sample duplicate	One every 20 project samples, or per SDG, whichever is more	RPD < 20%, or difference <CRDL when both	Examine the project-specific DQOs. Contact the client as to additional	For the specific analyte(s) in the parent sample, apply J if acceptance criteria are not met	The data shall be evaluated to determine the source of difference.

Table 7-D
Quality Control Requirements for Trace Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (Method 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Data Validation Flagging Criteria	Comments
	frequent, per matrix	results<5CRDL(between MS and MSD or sample and sample duplicate)	measures to be taken.	(RPD<50% for aqueous and RPD<100% for soil; difference<CRDL for aqueous and difference<2CRDL for soil when both concentrations <5CRDL).	
Internal standards (IS)	Every sample	IS intensity within 60-125% of intensity of the IS in the calibration blank	Original samples should be diluted by a factor of two, internal standards added, and the sample re-analyzed. Report the results of reanalysis if the internal standard responses are within the limits. If internal standard responses are still not within limits, note in SDG Narrative and report the results of the undiluted original sample analysis.	Flagging criteria is not appropriate.	No samples should be reported without passing internal standards.
Results reported between IDL and CRDL	NA	NA	NA	Apply J to all results between IDL and CRDL.	

Table 7-E
Quality Control Requirements for Perchlorate by Ion Chromatography (Method 314.0)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Initial Demonstration of Capability (IDC)	Prior to beginning any analysis batch.	See Table 5 of Method 314.0.	NA	
Matrix Conductivity Threshold (MCT)	As part of the initial demonstration of capability. See section 9.2.8 of Method 314.0.	MCT, based on linear regression, is the matrix conductance for which the peak area-to-height ratio percent difference exceeds 20%. See Table 5 of Method 314.0.	NA	
Method Detection Limit (MDL)	An MDL study is conducted at initial setup and subsequently once per 12-month period and when major changes occur in the methods operating procedures (addition of cleanup procedures, column changes, mobile phase changes). If no changes have been made to the method, quarterly MDL verification checks may be performed in lieu of the yearly MDL study.	MDL study must be performed in the matrix of interest using a standard at a concentration that is 1 to 10 times the estimated MDL value. MDL must be validated through the analysis of a low-level spike at ~ 2 times MDL taken through the entire preparation process. MDL verification checks must produce a signal at least 3 times the instrument's noise level.	Run MDL verification check at higher level and set MDL higher or perform the MDL study again.	Samples cannot be analyzed without a valid MDL.
Limit of Quantitation (LOQ; called MRL, Method Reporting Level in 314.1)	With every initial calibration.	Documented in the specific matrix of concern, at or below the applicable regulatory limit. Equal to lowest calibration standard. At least 3 times the MDL/LOD. The LOQ must be verified in a solution prepared at the MCT.	Flag all results between LOD and LOQ as "J".	

Table 7-E
Quality Control Requirements for Perchlorate by Ion Chromatography (Method 314.0)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Retention-Time (window width calculated for each analyte and internal standard)	At method setup and after major maintenance (e.g., column change).	Width is + 3 times standard deviation for each analyte retention time from 72-hour study.	NA	
Holding time (HT)	Applies to all samples.	HT < 28 days (to be consistent with other EPA requirements).	All data analyzed outside the required holding time should be qualified using professional judgment. If holding time is only slightly exceeded, data may be qualified with "J" or not at all. If holding time is grossly exceeded, data should be rejected (flagged as "R").	
Initial Calibration (ICAL)	Initial calibration prior to sample analysis.	Minimum of 5 calibration standards to establish linearity (daily), r2 > 0.995.	Correct problem, then repeat initial calibration. Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second Source Calibration Verification (SSCV)	Once after each multipoint calibration.	Value of second source for perchlorate within + 10% of expected value (initial source).	Correct problem and verify second source standard. Rerun SSCV. If that fails, correct problem and repeat initial calibration.	Problem must be corrected. No samples may be run until SSCV has passed.

Table 7-E
Quality Control Requirements for Perchlorate by Ion Chromatography (Method 314.0)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Initial Calibration Verification Standard (ICV)	After initial calibration, with each analysis batch, analysis of a standard at the LOQ	Recovery must be 85-115% of true value. Note: Method 314.0 requires + 25%; however, the DoD-QSM requires the acceptance criteria for the ICV to be the same as the continuing calibration verifications. As the QSM requirements are more stringent, they supersede the method requirements.	Correct problem and rerun ICV. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. No samples may be run until calibration has been verified.	Problem must be corrected. No samples may be run until calibration has been verified.
Instrument Performance Check (IPC)	One per analytical batch .Analysis of a standard containing mid-level perchlorate and interfering anions bracket each analytical batch to verify method performance at the matrix conductivity threshold. At least one IPC must be analyzed daily.	IPC conductance within + 10% of original measured value. Peak area-to-height ratio percent difference < 20% (compared to peak area-to-height ratio of the LCS). Perchlorate quantitated between 80 and 120% of fortified level. < 5% shift in perchlorate retention time.	Correct problem and then reanalyze all samples in that batch. If poor recovery from the cleanup filters is suspected, a different lot of filters must be used to re-extract all samples in the batch. If column degradation is suspected, a new column must be calibrated before the samples can be reanalyzed.	No samples may be reported as associated with a failing IPC.
Continuing Calibration Verification Standard (CCV)	Alternate analysis of mid-level standard and a standard at the LOQ after every 10 samples. At the end of the batch, both standards should be analyzed. All samples should be bracketed by the analysis of a standard demonstrating that the system was capable of accurately detecting and quantifying perchlorate.	Recoveries must fall between 85 and 115%.	Correct problem and rerun CCV and all samples analyzed since last successful CCV. If that fails, apply Q-flag to all results in all samples since the last acceptable calibration verification, if reanalysis is not possible.	No samples may be analyzed until the problem has been corrected.

Table 7-E
Quality Control Requirements for Perchlorate by Ion Chromatography (Method 314.0)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Method Blank or Pretreated Laboratory Reagent Blank	One per batch (up to 20 samples). Must undergo same pretreatment process that was performed on the samples.	<½ of the RL	Correct problem, re-prep, then reanalyze method blank and all samples processed with the contaminated blank. Apply B-flag to all results for the specific analytes in all samples in the associated preparatory batch if reanalysis is unsuccessful.	N/A
Pretreated QC	REQUIRED once per analytical batch if batch includes samples that have exceeded the MCT and have been pretreated in any way to reduce the common anion levels. Pretreated method blanks, LCS, ICS, and matrix spikes should be analyzed if any samples in the batch have required pretreatment to reduce common anions.	Apply criteria as stated above for individual QC elements.	Use corrective action/flagging criteria as stated above for individual QC elements.	Pretreated samples must have associated pretreated QC samples.
Laboratory Control Sample (LCS)	Once per analytical batch following the ICV. Calculate %Recovery prior to analyzing samples.	%Recovery within 85-115%.	Correct problem, then re-prep and reanalyze the LCS and all associated samples. If corrective action fails, all data should be rejected (flagged as "R")	Sample results from batches that fail the LCS are invalid.

Table 7-E
Quality Control Requirements for Perchlorate by Ion Chromatography (Method 314.0)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Matrix Spikes (MS)	One per 20 samples per matrix.	%Recovery within 80-120%.	No action is taken based upon MS/MSD data alone. However, using informed professional judgment, the MS/MSD results may be used in conjunction with other QC criteria to determine the need for qualification of the data.	For matrix evaluation only. If MS results are outside the limits, the data must be evaluated to determine the source of the difference and to determine if there is a matrix effect or analytical error.
Matrix Spike Duplicates or Laboratory Duplicates (MS and MSD)	one per 20 samples per matrix.	%Recovery within MS limits, RPD < 15%.	Flag all positive sample results as "J".	Evaluate the data to determine the source of the difference.

Table 7-F
Quality Control Requirements for Perchlorate by MS Methods (Method EPA 331.0, EPA 332.0, SW6850)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Holding Time (HT)	All samples	HT < 28 days	All data analyzed outside the required holding time should be qualified using professional judgment. If holding time is only slightly exceeded, data may be qualified with "J" or not at all. If holding time is grossly exceeded, data should be rejected (flagged as "R").	No criteria exist.
Limit of Quantitation (LOQ)	With every initial calibration.	Documented in the specific matrix of concern, at or below the applicable regulatory limit. Equal to lowest calibration standard. At least 3 times the MDL/LOD	Apply J-flag to all results between LOD and LOQ.	
Method Detection Limit (MDL)	A full MDL study is conducted at initial setup and subsequently once per 12-month period and when major changes occur in the method's operating procedures (addition of cleanup procedures, column changes, mobile phase changes). If no changes have been made to the method, quarterly MDL verification checks may be performed in lieu of the yearly MDL study.	MDL study must be performed in the matrix of interest using a standard at a concentration that is 1 to 10 times the estimated MDL value. MDL must be validated through the analysis of a low-level spike at ~ 2 times MDL taken through the entire preparation process. MDL verification checks must produce a signal at least 3 times the instrument's noise level.	Run MDL verification check at higher level and set MDL higher or re-conduct MDL study.	Samples cannot be analyzed without a valid MDL.

Table 7-F
Quality Control Requirements for Perchlorate by MS Methods (Method EPA 331.0, EPA 332.0, SW6850)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Retention Time (window width calculated for each analyte and internal standard)	At method setup and after major maintenance (e.g., column change).	Width is + 3 times standard deviation for each analyte retention time from 72-hour study.	N/A	N/A
Initial Calibration (ICAL)	Initial calibration prior to sample analysis.	Minimum of 5 calibration standards to establish linearity (daily), r2 > 0.995. The calibration is linear and shall not be forced through the origin.	Correct problem, then repeat initial calibration. Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Initial Calibration Verification Standard (ICV)	After initial calibration, daily analysis of a second source standard at the midpoint of the calibration.	%Difference < 15% relative to initial value.	Correct problem and rerun ICV. If that fails, correct problem and repeat initial calibration. Flagging criteria are not appropriate. No samples may be run until calibration has been verified.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing Calibration Verification Standard (CCV)	Analysis of mid-level standard after every 10 samples. All samples should be bracketed by the analysis of a standard, demonstrating that the system was capable of accurately detecting and quantifying perchlorate.	%Difference < 15% relative to initial value.	Correct problem and rerun CCV and all samples analyzed since last successful CCV. Flagging criteria are not appropriate. No samples may be run until calibration has been verified.	No samples may be analyzed until the problem has been corrected.

Table 7-F
Quality Control Requirements for Perchlorate by MS Methods (Method EPA 331.0, EPA 332.0, SW6850)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Method Detection Limit Verification Standard (MDLV)	Analysis of a standard containing perchlorate at 2 times the MDL concentration. This standard must be analyzed before and directly after every batch of samples is analyzed. It can be analyzed after every 10 samples in order to reduce the reanalysis rate.	Recovery within 30% of its true value.	Correct problem and rerun MDLV and all samples analyzed since last successful MDLV. Flagging criteria are not appropriate. No samples may be run until calibration has been verified.	No samples may be analyzed until the problem has been corrected.
Interference Check Sample (ICS)	Analysis of a standard containing perchlorate at the RL and interfering anions at the concentration determined by the interference threshold study. One ICS is extracted with every batch of 20 samples. It verifies the method performance at the matrix conductivity threshold (MCT). At least one ICS must be analyzed daily.	Monitor recovery of perchlorate and retention time. Recovery within 30%.	Correct problem and then reanalyze all samples in that batch. If poor recovery from the cleanup filters is suspected, a different lot of filters must be used to re-extract all samples in the batch. If column degradation is suspected, a new column must be calibrated before the samples can be reanalyzed.	No samples may be reported that are associated with a failing ICS.

Table 7-F
Quality Control Requirements for Perchlorate by MS Methods (Method EPA 331.0, EPA 332.0, SW6850)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Method Blanks (MB)	One per batch. Undergoes same pretreatment steps as the samples.	< ½ of the RL.	Correct problem, re-prep, then reanalyze method blank and all samples processed with the contaminated blank. If reanalysis fails criteria, flag all positive sample results within 5x the method blank concentration as "J".	
Laboratory Control sample (LCS)	Once per analytical batch spiked at the RL. Undergoes same pretreatment steps as the samples.	Recovery within method requirements or laboratory-generated limits, or 85-115% to verify calibration and to check method performance.	Correct problem, then re-prep and reanalyze the LCS and all associated samples. If corrective action fails, all data should be rejected (flagged as "R")	
Matrix Spikes (MS)	Collect one per 20 samples per matrix, spiked at the RL. Undergoes same pretreatment steps as the samples.	Recovery within 75-125%.	No action is taken based upon MS/MSD data alone. However, using informed professional judgment, the MS/MSD results may be used in conjunction with other QC criteria to determine the need for qualification of the data.	For matrix evaluation only. If MS results are outside the limits, the data must be evaluated to determine the source of the difference and to determine if there is a matrix effect or analytical error.
Matrix Spike Duplicates or Laboratory Duplicates (MS and MSD)	Collect one per 20 samples per matrix, spiked at the RL. Undergoes same pretreatment steps as the samples.	Recovery within MS limits, RPD < 20%.	Flag all positive sample results as "J".	Evaluate the data to determine the source of the difference.

Table 7-F
Quality Control Requirements for Perchlorate by MS Methods (Method EPA 331.0, EPA 332.0, SW6850)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Laboratory Reagent Blank	Analyzed prior to calibration and after samples with over-range concentration of perchlorate and after each batch is analyzed.	Concentration < ½ RL.	Reanalyze reagent blank (until no carryover is observed) and all samples processed since the contaminated blank. Apply J flag to all results not preceded by an acceptable reagent blank if reanalysis is not possible.	
QC Criteria Specific to MS Confirmation				
Mass Tuning	Daily before sample analysis.	Tuning standards should contain the analytes of interest.	Retune instrument. If the tune will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Sample analysis should not proceed without an acceptable tuning.
Mass Calibration	Performed prior to sample analysis and calibration curve analysis.	Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used and the same mass calibration must be used for all data files in an analytical run. Acceptance criteria must be clearly stated in the laboratory's SOP.	If the mass calibration fails, recalibrate. If it still fails, consult manufacturer instructions on corrective maintenance.	No samples may be analyzed under a failing mass calibration.
Isotope Ratio 35Cl/37Cl	Every sample, spiked sample, and standard and method blank.	Monitor for both the parent ion at mass 99/101 and the product ion at mass 83/85 for MS-MS methods or just 99/101 for MS only. Theoretical ratio ~ 3.06. Must fall between 2.2 to 3.3.	If criteria are not met, the sample must be rerun. If the sample was not pretreated, the sample should be re-extracted using cleanup procedures. If, after cleanup, the ratio still fails, use alternative techniques to confirm presence of perchlorate (i.e., a post spike sample, dilution to reduce any interferences, etc.). Data should be qualified as estimated and should be noted in the case narrative.	Decision to report data failing ratio check should be thoroughly documented in case narrative.

Table 7-F
Quality Control Requirements for Perchlorate by MS Methods (Method EPA 331.0, EPA 332.0, SW6850)

QC Element	Minimum Frequency	Criteria/Requirements	Corrective Action/ Flagging Criteria	Comments
Internal Standard (IS)	Addition of 18O-labeled perchlorate to every sample, spiked sample, standard, instrument blank, and method blank.	Measured 18O IS area within + 50% of the value from the initial calibration (retention time window of ~ 0.3% for perchlorate and IS).	Rerun the sample at increasing dilutions until the + 50% acceptance criteria are met. If criteria cannot be met with dilution, the interferences are suspected and the sample must be re-prepped using further pretreatment steps. Data should be qualified as estimated with a J flag and should be discussed in the case narrative.	If peak is not within retention time window, presence is not confirmed. Use for quantitation and to ensure identification. Failing internal standard should be thoroughly documented in the case narrative.
Interference Threshold Study	At initial setup and when major changes occur in the method's operating procedures (addition of cleanup procedures, column changes, mobile phase changes).	Measure the threshold of common suppressors (chloride, sulfate, carbonate, bicarbonate) that can be present in the system without affecting the quantitation of perchlorate. The threshold is the concentration of the common suppressors where perchlorate recovery falls outside of a 90-110% window.	N/A	This study and site history will determine the concentration at which the ICS suppressors

Table 8
Surrogate Recovery Limits

Analyte	Water (1)		Soil (2)	
	Lower Control Limit (%)	Upper Control Limit (%)	Lower Control Limit (%)	Upper Control Limit (%)
CLP VOCs (Multi Level)				
Toluene-d8 (TOL)	88	110	84	138
Bromofluorobenzene (BFB)	96	115	59	113
1,2 dichloroethane-d4 (DCE)	76	114	70	121
CLP VOCs (Low Concentration, Method 524.2)				
Vinyl Chloride-d3	49	138		
Chloroethane-d5	60	126		
1,1-Dichloroethene-d2	65	130		
2-Butanone-d5	42	171		
Chloroform-d	80	123		
1,2-Dichloroethane-d4	78	129		
Benzene-d6	78	121		
1,2-Dichloropropane-d6	84	123		
Toluene-d8	77	120		
trans-1,3-Dichloropropene-d4	80	128		
2-Hexanone-d5	37	169		
Bromoform-d	76	135		
1,1,2,2-Tetrachloroethane-d2	75	131		
1,2-Dichlorobenzene-d4	50	150		
CLP SVOCs (Multi Level)				
Nitrobenzene-d5 (Base/Neutral)	35	114	23	120
2-Fluorobiphenyl (Base/Neutral)	43	116	30	115
Terphenyl- d14 (Base/Neutral)	33	141	18	137
Phenol-d5 (Acid)	10	110	24	113
2-Fluorophenol (Acid)	21	110	25	121
2,4,6 Tribromophenol (Acid)	10	123	19	122
2-Chlorophenol- d4 (Acid)	33 (advisory)	110 (advisory)	20 (advisory)	130 (advisory)
1,2-Dichlorobenzened4	16 (advisory)	110 (advisory)	20 (advisory)	130 (advisory)
CLP SVOCs (Low Concentration)				
Phenol-d5	10	110		
bis-(2-Chloroethyl)ether-d8	41	94		
2-Chlorophenol-d4	33	110		
4-Methylphenol-d8	38	95		
Nitrobenzene-d5	35	114		
2-Nitrophenol-d4	40	106		
2,4-Dichlorophenol-d3	42	98		
4-Chloroaniline-d4	8	70		
Dimethylphthalate-d6	62	102		
Acenaphthylene-d8	49	98		
4-Nitrophenol-d4	9	181		
Fluorene-d10	50	97		

Table 8
Surrogate Recovery Limits

Analyte	Water (1)		Soil (2)	
	Lower Control Limit (%)	**Upper Control Limit (%)**	**Lower Control Limit (%)**	**Upper Control Limit (%)**
4,6-Dinitro-2-methylphenol-d2	53	153		
Anthracene-d10	55	116		
Pyrene-d10	47	114		
Benzo(a)pyrene-d12	54	120		
CLP Pesticides/PCBs				
Decachlorobiphenyl	30 (advisory)	150 (advisory)	30 (advisory)	150 (advisory)
Tetrachloro-m-xylene	30 (advisory)	150 (advisory)	30 (advisory)	150 (advisory)
SW846 Method 8330				
TBD[(2)]	50	150	50	150

Notes:
(1) For CLP VOC/SVOC/Pesticides/PCB, surrogate recoveries from OLM04.3 (March 2003), Exhibit D. For low concentration level aqueous samples, additional surrogates are required for VOC/SVOC analysis and see specific requirements described in OLC03.2 (December 2000).
(2) Site-specific work plan must specify which surrogate compound is intended.

Table 9-A
LCS Control Limits For Explosives
SW-846 Method 8330
Water Matrix

Analyte	Lower Control Limit (%)	Upper Control Limit (%)
1,3,5-Trinitrobenzene	65	140
1,3-Dinitrobenzene	45	160
2,4-Dinitrotoluene	60	135
2,6-Dinitrotoluene	60	135
2,4,6-Trinitrotoluene (TNT)	50	145
2-Amino-4,6-dinitrotoluene	50	155
2-Nitrotoluene	45	135
3-Nitrotoluene	50	130
4-Amino-2,6-dinitrotoluene	55	155
4-Nitrotoluene	50	130
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	50	160
Methyl-2,4,6-trinitrophyenylnitramine (Tetryl)	20	175
Nitrobenzene	50	140
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	80	115
Nitroglycerin	60	120
Pentaerythritol Tetranitrate	60	120

Table 9-B
LCS Control Limits For Explosives
SW-846 Method 8330
Solid Matrix

Analyte	Lower Control Limit (%)	Upper Control Limit (%)
1,3,5-Trinitrobenzene	75	125
1,3-Dinitrobenzene	80	125
2,4-Dinitrotoluene	80	125
2,6-Dinitrotoluene	80	120
2,4,6-Trinitrotoluene (TNT)	55	140
2-Amino-4,6-dinitrotoluene	80	125
2-Nitrotoluene	80	125
3-Nitrotoluene	75	120
4-Amino-2,6-dinitrotoluene	80	125
4-Nitrotoluene	75	125
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	70	135
Nitrobenzene	75	125
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	75	125
Nitroglycerin	60	120
Pentaerythritol Tetranitrate	60	120

Table 9-C
LCS Control Limits for Organochlorine Pesticides

CLP OLC03.0, SW-846 Method 8081A Water Matrix		
Analyte	**Lower Control Limit (%)**	**Upper Control Limit (%)**
4,4'-DDE	50	150
Dieldrin	30	130
Endosulfan sulfate	50	120
Endrin	50	120
gamma-BHC	50	120
gamma-Chlordane	30	130
Heptachlor epoxide	50	150
CLP OLM04.3, SW-846 Method 8081A Water/Soil Matrix		
All analytes	Laboratory advisory limits, or 70~130% when not available	

Notes:
1) For low concentration organic analysis, limits based on Table D-3 from Draft OLC03.0, December 2000.
2) For multi-media, multi-concentration organic analysis, laboratory advisory limits or 70~130% (when laboratory limits not available) will be used in accordance with the USEPA Region 2 SOP for Method 8082.

Table 9-D
LCS Control Limits for Polychlorinated Biphenyls
SW-846 Method 8082
Water/Soil Matrix

Analyte	Lower Control Limit (%)	Upper Control Limit (%)
Aroclor 1016	Laboratory advisory limits, or 70~130% when not available	
Aroclor 1260	Laboratory advisory limits, or 70~130% when not available	

Note:
Laboratory advisory limits or 70~130% (when laboratory limits not available) will be used in accordance with the USEPA Region 2 SOP for Method 8082.

Table 9-E
LCS Control Limits for Inorganics
SW-846 Methods 6010B, 6020, 7470A, and 7580
Water Matrix

Analyte	Lower Control Limit (%)	Upper Control Limit (%)
Aluminum	80	120
Antimony	80	120
Arsenic	80	120
Barium	80	120
Beryllium	80	120
Cadmium	80	120
Calcium	80	120
Chromium	80	120
Cobalt	80	120
Copper	80	120
Iron	80	120
Lead	80	120
Magnesium	80	120
Manganese	80	120
Mercury	80	120
Molybdenum	80	120
Nickel	80	120
Potassium	80	120
Selenium	80	120
Silver	80	120
Sodium	80	120
Strontium	80	120
Thallium	80	120
Titanium	80	120
Vanadium	80	120
White Phosphorus	75	125
Zinc	80	120
Zirconium	80	120

Table 9-F
LCS Control Limits for Inorganics
SW-846 Methods 6010B, 6020, 7471A, and 7580
Solid Matrix

Analyte	Lower Control Limit (%)	Upper Control Limit (%)
Aluminum	80	120
Antimony	80	120
Arsenic	80	120
Barium	80	120
Beryllium	80	120
Cadmium	80	120
Calcium	80	120
Chromium	80	120
Cobalt	80	120
Copper	80	120
Iron	80	120
Lead	80	120
Magnesium	80	120
Manganese	80	120
Mercury	80	120
Molybdenum	80	120
Nickel	80	120
Potassium	80	120
Selenium	80	120
Silver	75	120
Sodium	80	120
Strontium	80	120
Thallium	80	120
Titanium	80	120
Vanadium	80	120
White Phosphorus	75	125
Zinc	80	120
Zirconium	80	120

Table 10-A
MS/MSD Control Limits for Volatiles

CLP OLC03.2/OLM04.3, SW-846 Method 8260B Water Matrix			
Analyte	**Lower Control Limit (%)**	**Upper Control Limit (%)**	**RPD%**
1,1-Dichloroethene	61	145	14
Benzene	76	127	11
Trichloroethene	71	120	14
Toluene	76	125	13
Chlorobenzene	75	130	13
CLP OLM04.3, SW-846 Method 8081A Soil Matrix			
1,1-Dichloroethene	59	172	22
Benzene	66	142	21
Trichloroethene	62	137	24
Toluene	59	139	21
Chlorobenzene	60-	133	21

Notes:
1) For water matrix, limits based on Table D-6 from Draft OLC03.0, December 2000 and Table 8 in Exhibit D from OLM04.3, March 2003.
2) For soil matrix, limits based on Table 8 in Exhibit D from OLM04.3, March 2003.

Table 10-B
MS/MSD Control Limits for Semivolatiles

CLP OLC03.2/OLM04.3, SW-846 Method 8270C Water Matrix			
Analyte	**Lower Control Limit (%)**	**Upper Control Limit (%)**	**RPD%**
Phenol	12	110	42
2-Chlorophenol	27	123	40
N-Nitroso-di-n-propylamine	41	116	38
4-Chloro-3-methylphenol	23	97	42
Acenaphthene	46	118	31
4-Nitrophenol	10	80	50
2,4-Dinitrotoluene	24	96	38
Pentachlorophenol	9	103	50
Pyrene	26	127	31
CLP OLM04.3, SW-846 Method 8270C Soil Matrix			
Phenol	26	90	35
2-Chlorophenol	25	102	50
N-Nitroso-di-n-propylamine	41	126	38
4-Chloro-3-methylphenol	26	103	33
Acenaphthene	31	137	19
4-Nitrophenol	11	114	50
2,4-Dinitrotoluene	28	89	47
Pentachlorophenol	17	109	47
Pyrene	35	142	36

Notes:
1) For water matrix, limits based on Table D-6 from Draft OLC03.0, December 2000 and Table 6 in Exhibit D from OLM04.3, March 2003.
2) For soil matrix, limits based on Table 6 in Exhibit D from OLM04.3, March 2003.

Table 10-C
MS/MSD Control Limits for Organochlorine Pesticides

CLP OLC03.2/OLM04.3, SW-846 Method 8081A Water Matrix			
Analyte	**Lower Control Limit (%)**	**Upper Control Limit (%)**	**RPD%**
4,4'-DDT	38	127	27
Aldrin	40	120	22
Dieldrin	52	126	18
Endrin	56	121	21
gamma-BHC	56	123	15
Heptachlor	40	131	20
CLP OLM04.3, SW-846 Method 8081A Soil Matrix			
4,4'-DDT	23	134	50
Aldrin	34	132	43
Dieldrin	31	134	38
Endrin	42	139	45
gamma-BHC	46	127	50
Heptachlor	35	130	31

Notes:
1) For water matrix, limits based on Table D-3 from Draft OLC03.0, December 2000 and Table 3 in Exhibit D from OLM04.3, March 2003.
2) For soil matrix, limits based on Table 3 in Exhibit D from OLM04.3, March 2003.

TABLE 11
Field Equipment Calibration and Maintenance, Testing and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptable Criteria	Corrective Action	Responsible Party	SOP Reference Number
Horiba U-22 Water Quality Meter Dissolved Oxygen	Manual calibration with 2 standards (zero and saturated)	Replace internal solution (monthly); Clean probes before storage. Replace DO Membrane. Replace Battery		Inspect sponge and replace as necessary (storage)	Morning and Evening	+/- 0.2 mg/L for 0.0 mg/L DO Standard	Replace DO membrane on probe.	Field Team Leader	See Appendix B of FSP for Manual
Horiba U-22 Water Quality Meter ORP	Manual calibration using standard solution	Replace Battery			Morning and Evening	Within 15 mV of solution	Recalibrate; Replace electrode or have meter inspected	Field Team Leader	See Appendix B of FSP for Manual
Horiba U-22 Water Quality Meter Temperature	Manual calibration using known temperature standard	Replace Probe; Replace Battery			Morning and Evening	+ / – 2 degrees	Recalibrate	Field Team Leader	See Appendix B of FSP for Manual
Horiba U-22 Water Quality Meter Conductivity	Auto calibration using a 4 pH standard solution	Replace Battery			Morning and Evening	+ /– 5%	Clean contacts on probe per owners manual	Field Team Leader	See Appendix B of FSP for Manual
Horiba U-22 Water Quality Meter pH	Auto calibration using a 4 pH standard solution	Replace Battery			Morning and Evening	+ / – 5%	Clean contacts on probe per owners manual; recalibrate with new	Field Team Leader	See Appendix B of FSP for Manual

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptable Criteria	Corrective Action	Responsible Party	SOP Reference Number
							solution		
MiniRae PID	Calibration with 2 points (zero and standard)	Clean PID Lamp and Filter (when zero creeps upward or when in contact with moisture); check battery		Inspect filter for dust/foreign objects	Morning and Evening	+/- 2 ppm	Recalibrate	Field Team Leader	See Appendix B of FSP for Manual
Horiba U-22 Water Quality Meter Turbidity	Auto calibration using a 4 pH standard solution	Clean Lens, Replace Battery	Standard Check		Morning and Evening	< / = 5ntu	Recalibrate	Field Team Leader	See Appendix B of FSP for Manual
HACH Digital Titrator	Standard Check	Replace Reagent	Standard Check		Morning and Evening	< / = 5	Rerun standard check	Field Team Leader	See Appendix B of FSP for Manual
HACH Colorimeter	Standard Check	Replace battery	Standard Check		Morning and Evening	< / = 5	Rerun standard check	Field Team Leader	See Appendix B of FSP for Manual

Table 12
Performance Criteria for Field Duplicates and Laboratory Duplicates

Laboratory Duplicate		
	Frequency	RPD
Metals (ILM05.3)	One per SDG, or 20 field samples in a SDG, or a group of field samples of a similar concentration level.	20% or CRDL
MEE	One per SDG, or 20 field samples in a SDG, or a group of field samples of a similar concentration level.	20% or RL
VOC/SVOC/Pesticides/PCBs (Matrix Spike Duplicate)	One per SDG, or 20 field samples in a SDG, or a group of field samples of a similar concentration level.	See Table 10
Explosives	One per extraction batch.	20%
Perchlorate	One per SDG, or 20 field samples in a SDG, or a group of field samples of a similar concentration level.	15%
Field Duplicate		
VOC/SVOC/Pesticides/PCBs, TCLP VOC/SVOCs	One every 20 project samples, or per SDG, whichever is more frequent, per matrix	25% (water) 50% (soil)
Metals, TCLP Metals	One every 20 project samples, or per SDG, whichever is more frequent, per matrix	50% or CRDL (water) 100% or CRDL (soil)
MEE	10% of project samples	25% (water) 50% (soil)
Explosives	One every 20 project samples, or per SDG, whichever is more frequent, per matrix	25% (water) 50% (soil)
Perchlorate	One every 20 project samples, or per SDG, whichever is more frequent, per matrix	25% (water) 50% (soil)

TABLE 13
Inspection/Acceptance Testing Requirements for Consumables and Supplies

Critical Supplies/ Consumables	Inspection/ Acceptance Specifications	Acceptance Criteria	Testing Method	Responsible Party	Handling/ Storage Conditions	Vendor
Isobutylene	Ensure container is pressurized	Visual	Used for PID calibration	Field Team Leader	None specified	Pine Environmental[1]
4 pH standard solution	Inspect for contamination periodically	Visual, or based on poor calibration results	Used for Horiba U-22 Auto calibration	Field Team Leader	None specified	Pine Environmental[1]
Powder for ORP standard solution			Used for Horiba U-22 ORP calibration	Field Team Leader	None specified	Pine Environmental[1]
Sodium hydroxide standard solution	Check fluid monthly for strength	5 – 5.25 mL of solution	Used for CO_2 Hach Titrator	Field Team Leader	None specified	Pine Environmental[1]
Reagent water	Check for evidence of tampering	Visual		Field Team Leader	None specified	Contracted laboratory

Note:
1 – Pine Environmental is the primary vendor used by Parsons for field equipment; other vendors may be utilized for the project.

Table 14
Critical Supplies and Consumables Tracking Log

Tracking Number	Date Received	Meets Inspection/Acceptance Criteria (Y/N, if yes include date)	Retesting Needed (Y/N, if yes include date)	Expiration Date	Initials/Date

Table 15
Sample Handling System

Sample Collection, Packaging and Shipment	
Sample Collection	Field Team
Sample Packing	Field Team
Coordination of Shipment	Field Team Leader
Carrier	Federal Express or UPS - overnight
Sample Receipt and Analysis	
Sample Receipt	Field Analyst / Laboratory
Sample Custody	Field Analyst / Laboratory
Sample Preparation	Field Analyst / Laboratory
Sample Analysis	Field Analyst / Laboratory
Sample Archiving	
Field Sample Storage and Archive	Field Team
Sample Rinse Blanks	Field Team
Sample Trip Blanks	Field Team
Sample Duplicated	Field Team
Sample Disposal	
Sample Disposal	Field Analyst / Laboratory
Storage at Laboratory	Laboratory

Table 16
Summary of Calibration and QC Procedures for Screening Methods [a]

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action [b]	Data Flagging Criteria
USEPA Method 160.3	Percent Solids	Field Duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
SW846 Method 1010A/	Ignitability	Field Duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
SW846 Method 1020B/	Ignitability	Field Duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
SW846 Method 1030	Ignitability for soilds	Field Duplicate	10% of field samples	Burning rate within ± 10%	Correct problem, repeat measurement	J
SW846 Method 1110	Corrosivity	Duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
SW846 Method 9040C with commercially available pH meter	pH (water)	2-point calibration required for each instrument	Once per day	± 0.05 pH units for every buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration	Flagging criteria not appropriate
		pH 7 buffer	At each sample location	± 0.1 pH units	Correct problem, recalibrate	Flagging criteria not appropriate
		Field Duplicate	10% of field samples	± 0.1 pH units	Correct problem, repeat measurement	J
SW846 Method 9045D	pH (soil)	2-point calibration required for each instrument	Once per day	± 0.05 pH units for every buffer	Check with new buffer; if still out, reapir meter, repeat calibration check	Flagging criteria not appropriate
		pH 7 buffer	At each sample location	± 0.1 pH units	Recalibrate	Flagging criteria not appropriate
		Field Duplicate	10% of field samples	± 0.1 pH units	Correct problem, repeat measurement. If still out, repeat calibration and reanalyze samples	J

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action [b]	Data Flagging Criteria
SW846 Method 9050A with commercially available conductivity meter	specific conductance	Calibration with KCl standard	Once per day at beginning of testing	± 5%	If calibration is not achieved, check meter, standards, and probe; repeat calibration	Flagging criteria not appropriate
		Field Duplicate	10% of field samples	± 5%	Correct problem, repeat measurement	J
SW846 Method 9060A/USEPA Method 415.1/Lloyd Kahn	Total Organic Carbon	Method Blank	one per batch	≤RL	Clean system, reanalyze blank. Repeat until <RL	J
		Independent Standard	one per 15 samples	RPD < 20%	Correct problem, repeat measurement	J
		Spike Duplicate	one per 10 samples	RPD < 20%	Repeat measurement	J
USEPA Method 130.1	Hardness	Field Duplicate	10% of field samples	Burning rate within ± 10%	Correct problem, repeat measurement	J
USEPA Method 170.1 with commercially available thermometer	Temperature	Field Duplicate	10% of field samples	± 1.0 °C	Correct problem, repeat measurement	J
USEPA Method 180.1 with commercially available turbidity meter	Turbidity	Calibration with one formazin standard per instrument range used	Once per day at beginning of test	± 5 units, 0-100 range ± 0.5 units, 0-0.2 range ± 0.2 units, 0-1 range	If calibration is not achieved, check meter; replace if necessary, recalibrate	Flagging criteria not appropriate
		Field Duplicate	10% of field samples	RPD ≤ 20%	Correct problem, repeat measurement	J
E310.1/Hach Method 8203 or similar	Alkalinity	Field Duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
E360.1	Dissolved oxygen	Field Duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
Organic Vapor Analysis	Hydrocarbon vapor	Samples with elevated readings will be submitted for definitive analysis.				

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action [b]	Data Flagging Criteria
USEPA Method 410.1	Chemical Oxygen Demand	Field Duplicate	10% of field samples	RPD < 20%	Correct problem, repeat measurement	J
		Method Blank	one per batch	≤RL	Clean system, reanalyze blank. Repeat until < RL	J
USEPA 351.2 / SM4500	Total Kjeldahl Nitrogen (TKN)	Linear Calibration Range using three standards and a blank	every 6 months or significant change in instrument response	± 10% linearity	Reestablish linearity, if range shown to be nonlinear, use sufficient standard to clearly define nonlinear portion.	Flagging criteria not appropriate
		Quality Control Sample	At the beginning of test (quarterly), or as required for data-quality	Concentrations ±10% of stated values	Correct problem, reanalyze QCS. Repeat until concentrations within ± 10% of stated values	Flagging criteria not appropriate
		Method Detection Limit	every 6 months, when a new operator begins work, or significant change in instrument response	-		Flagging criteria not appropriate
		Laboratory Reagent Blank	one per batch of samples	< MDL	Correct problem, repeat measurement	J
		Laboratory Fortified Blank	one per batch of samples	Recovery between 90-110%	Correct problem, repeat measurement	J
		Quality Control Sample	At the beginning of test (quarterly), or as required for data-quality	Concentrations ±15% of stated values	Correct problem, reanalyze QCS. Repeat until concentrations within ± 10% of stated values	Flagging criteria not appropriate
		Method Detection Limit	every 6 months, when a new operator begins work, or significant change in instrument response	-		Flagging criteria not appropriate
		Laboratory Reagent Blank	one per batch of samples	< MDL	Correct problem, repeat measurement	J

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action [b]	Data Flagging Criteria
		Laboratory Fortified Blank	one per batch of samples	Recovery between 75-125% (MRL to 10xMRL), between 85-115% (> 10xMRL)	Correct problem, repeat measurement	J
		Instrument Performance Check Solution	Checked daily	Peak Gaussian Factor between 0.8 and 1.15 to demonstrate proper instrument performance.	Retention times with a > 2% shift should be investigated and corrected. If column retention time noticeably shifts < 80% from original values, it should be cleaned or replaced.	J
		Spike Duplicate	10% of field samples	Recovery between 75-125%	Investigate if matrix influences are a factor.	J
		Surrogate Recovery		Surrogate recovery between 90-115%	Correct problem, repeat measurement	J
		Field or Laboratory Duplicate	10% of field samples, or one per sample batch	RPD ≤ 20%	Correct problem, repeat measurement	J
Hach 8146/8034/8131/8205	Ferrous iron/manganese/sulfide/carbon dioxide	In accordance with Hach Method Reference. Field duplicate to be collected for 10% of field sampls and RPD should be within 20%.				

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action [b]	Data Flagging Criteria
ASTM D1498 with commercially available ORP instrument	Oxidation-reduction potential	Sensitivity verification	Daily	ORP should decrease when pH is increased	If ORP increases, correct the polarity of electrodes. If ORP still does not decrease, clean electrodes and repeat procedure.	Flagging criteria not appropriate
		Calibration with one standard.	Daily.	Two successive readings ±10 millivolts	Correct problem, recalibrate	Flagging criteria not appropriate
		Field Duplicate	10% of field samples	±10 millivolts	Correct problem, repeat measurement	J

Notes:
a. If commercial instrument is used for the analysis, the calibration and QA/QC procedure shall be consistent with the manufacturer's manual or reference.
b. All corrective actions shall be documented, and the records shall be maintained by Parsons.

Table 17-A. Inorganic Data Qualifier Flags

Data Qualifier	Definition
J	The associated value is an estimated quantity.
UJ	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
R	The data was unusable. (Note: Analyte may or may not be present.).
U	The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.

Table 17-B. Organic Data Qualifier Flags

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation 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.

Table 18

An Example of SVOC Data Validation Sheet

PROJECT NAME/NO.

SDG:

Lab:

MEDIA:

Fraction: SVOC

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, & Solids Percentage				
System Monitoring Compounds				
Matrix Spike/Matrix Spike Duplicates				
Blanks				
GC/MS Instrument Performance Check				
TCL Analytes				
Tentatively Identified Compounds				
Reported Quantitation Limits				
GC/MS Initial Calibration				
GC/MS Continuing Calibration/Calibration Verification				
Internal Standards				
Field Duplicate				

Table 18

An Example of Pesticides Data Validation Sheet

PROJECT NAME/NO.
SDG:
Lab:
MEDIA: Soil
Fraction: Pesticides

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, & Solids Percentage				
System Monitoring Compounds				
Matrix Spike/Matrix Spike Duplicates				
Blanks				
GC Instrument Performance Check				
TCL Analytes				
Reported Quantitation Limits				
GC Initial Calibration				
GC Continuing Calibration/Calibration Verification				
Field Duplicate				

Note: GPC cleanup and sulfur cleanup were conducted for pesticides. No information of cleanup effectiveness was provided.
The lab indicated that all QC samples (LCS/blank/MS/MSD) went through the same clean-up as the samples.
It should be noted that the lower value of the values from the two columns was reported as the lab suspected interference.

Table 18

An Example of Metals Data Validation Sheet

PROJECT NAME/NO.
SDG:
FRACTION: metals
LAB:
MEDIA:

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				
Calibration				
Blanks (method blank, prep blank)				
Interference Check Sample				
CRDL Standard				
Laboratory Control Sample				
Duplicates				
Spike Sample Analysis				
ICP Serial Dilution				
Detection Limits				
ICP Linear Range				
Solids Percentage				

Table 19
SAP Distribution List

Personnel Name	Position/Project Title	Organization Name	Telephone Number	Fax Number	E-mail Address
Todd Heino	Program Manager	Parsons	617-449-1405	617-946-9777	todd.heino@parsons.com
Jeff Adams	Task/Project Manager	Parsons	617-449-1570	617-946-9777	jeff.adams@parsons.com
Jackie Travers	Task/Project Manager	Parsons	617-449-1566	617-946-9777	jacqueline.travers@parsons.com
Jim Lowerre	Quality Assurance Officer	Parsons	617-449-1559	617-946-9777	jim.lowerre@parsons.com
David Miller	Senior Customer Service Manager	Severn Trent Laboratories	412-963-7058	412-963-2468	dmmiller@stl-inc.com
Lisa Reyes	Quality Assurance Manager	Columbia Assurance Services	585-228-5380	585-288-8475	lreyes@rochester.caslab.com
Nancy Mattern	Quality Assurance Officer	General Engineering Laboratories	843-556-8171	843-766-1178	nancy.mattern@gel.com
Tony Bogolin	Project Manager	Severn Trent Laboratories, Inc., Buffalo	716-691-2600	716-691-7991	tbogolin@stl-inc.com
Tom Andrews	Field Team Leader	Parsons	716-633-7074	716-633-6195	Tom.Andrews@parsons.com
Tammy Chang	Project Chemist	Parsons	512-719-6092	512-719-6099	tammy.chang@parsons.com
Julio F. Vazquez	USEPA Region II Project Manager	USEPA Region II	212-637-4323	212-637-3256	vazquez.julio@epamail.epa.gov
Kuldeep K. Gupta	NYSDEC Project Manager	NYSDEC	518-402-9620		kxgupta@gw.dec.state.ny.us
Scott Bradley	Commander	USACE, Huntsville	256-895-1637	256-895-1602	Scott.G.Bradley@usace.army.mil
Keith Hoddinott	Commander	USACHPPM (PROV)	410-436-5209	410-436-5237	Keith.Hoddinott@amedd.army.mil

Personnel Name	Position/Project Title	Organization Name	Telephone Number	Fax Number	E-mail Address
Chris Boes	Commander	USACE, Aberdeen Proving Grounds	410-436-1513	410-436-1548	Christopher.boes@aec.apgea.army.mil
Edward Kashdan	Contractor for the USEPA	Gannett Fleming, Inc, Audubon, PA	610-650-8101	610-650-8190	
Steve Absolom	Commander's Representative	SEDA	607-869-1309	607-869-1362	stephen.m.absolom@us.army.mil
Randall Battaglia		USACE, NY District	607-869-1523	607-869-1251	randy.w.battaglia@nan02.usace.army.mil
Janet Fallo		USACE, NY District	607-869-1248	607-869-1251	Janet.R.Fallo@nan02.usace.army.mil
Charlotte Bethoney	Public Health Specialist	Bureau of Environmental Exposure Investigation	518-402-7850		
TBD	Field Subcontractor	TBD	TBD	TBD	TBD

Appendix F

NYSDEC Analytical Services Protocol CD

Appendix G

Parsons Standard Forms for Field Work

PARSONS
TEST PIT RECORD

Project Name: _____ TEST PIT NO.

Project Number: _____ Location: _____

Date / Time Start: _____

Date / Time Finish: _____

Weather: _____

Contractor: _____

Inspector(s): _____

DEPTH (ft bgs)	Stratigraphy	Macro	FIELD IDENTIFICATION OF MATERIAL	COMMENTS
_0				
___1				
___2				
___3				
___4				
___5				
___6				
___7				
___8				
___9				
___10				

EXCAVATION DIMENSIONS: (Length X Width X Depth) _____

AIR MONITORING DATA: Background OVM Reading: _____

Maximum Breathing Zone OVM Reading: _____

TIME	SAMPLE I.D.	LOCATION

Analysis Requested:

CROSS SECTION
(Include approximate dimensions)

OVERBURDEN BORING REPORT

PARSONS	CLIENT:	BORING NO.:

PROJECT : _____

SWMU # (AREA) : _____

SOP NO.:

START DATE: _____

FINISH DATE: _____

CONTRACTOR: _____

DRILLER: _____

INSPECTOR: _____

CHECKED BY: _____

CHECK DATE: _____

BORING CONVERTED TO MW? Y N

DRILLING SUMMARY

DRILLING METHOD	HOLE DIA.(ft)	DEPTH INTERVAL (ft)	SAMPLER SIZE	SAMPLER TYPE	HAMMER TYPE	HAMMER WT/FALL

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 (TEMP., WIND, ETC.)

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 METER		

INVESTIGATION DERIVED WASTE

DATE			
SOIL AMOUNT : (fraction of drum)			
DRUM #, LOCATION:			

COMMENTS:

SAMPLES TAKEN:

SAMPLES _____

DUPLICATES _____

MS/MSD _____

MRD

OVERBURDEN BORING REPORT

PARSONS	CLIENT:	BORING NO.:
COMMENTS:		DRILLER: _____
		INSPECTOR: _____
		DATE:

DEPTH (FT)	SAMPLING			SAMPLE				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
	BLOWS PER 6 INCHES	PENE-TRATION RANGE (FEET)	RECOV-ERY RANGE (FEET)	DEPTH INT (FEET)	NO.	VOC	RAD SCRN			

CORE BORING REPORT

PARSONS | **CLIENT:** USACOE | **BORING #:**

PROJECT: _____

SWMU # (AREA) : _____

SOP NO.:

DATE CORING STARTED: _____

DATE CORING COMPLETED: _____

CONTRACTOR: _____

DRILLER: _____

INSPECTOR: _____

GEOLOGIST: _____

CHECKED BY: _____

DATE CHECKED: _____

TOTAL FOOTAGE CORED: _____

OVERBURDEN THICKNESS: _____

GALLONS OF WATER USED

MONITORING

INTRUMENT	INTERVAL	BACKGROUND	TIME

COMMENTS:

CORE EQUIPMENT BARREL LENGTH (ft):

TYPE	SERIES	RANGE	O.D.	I.D.

DEPTH FEET	RUN # RANGE FEET	CORE RECOVERY FEET	MON. DATA	RQD %	SCHEMATIC STRATA/ FRACTURES	ANGLES DIP/STRIKE (BD,FL,JNT,FC)	BEDROCK/ CORE DESCRIPTIONS AND REMARKS (color, major modifiers, rock type, minor components, bedding or foliation, strike of joints/fractures relative to foliation, weathering on fractures, etc.)

INVESTIGATION DERIVED WASTE :

DATE			
SOIL AMOUNT (fraction of drum)			
DRUM #, LOCATION			

OVERBURDEN MONITORING WELL
COMPLETION REPORT & INSTALLATION DETAIL
PROTECTIVE RISER COMPLETION

PARSONS	CLIENT:	WELL #:

PROJECT: _____ PROJECT NO: _____

LOCATION: _____ INSPECTOR: _____

CHECKED BY: _____

DRILLING CONTRACTOR: _____ POW DEPTH: _____

DRILLER: _____ INSTALLATION STARTED: _____

DRILLING COMPLETED: _____ INSTALLATION COMPLETED: _____

BORING DEPTH: _____ SURFACE COMPLETION DATE: _____

DRILLING METHOD(S): _____ COMPLETION CONTRACTOR/CREW: _____

BORING DIAMETER(S): _____ BEDROCK CONFIRMED (Y/N?) _____

ASSOCIATED SWMU/AOC: _____ ESTIMATED GROUND ELEVATION: _____

PROTECTIVE SURFACE CASING:

DIAMETER: _____ LENGTH: _____ TOR: _____

RISER:

TOC: _____ TYPE: _____ DIAMETER: _____ LENGTH: _____

SCREEN: SLOT

TSC: _____ TYPE: _____ DIAMETER: _____ LENGTH: _____ SIZE: _____

POINT OF WELL: (SILT SUMP)

TYPE: _____ BSC: _____ POW: _____

GROUT:

TG: _____ TYPE: _____ LENGTH: _____

SEAL: TBS: _____ TYPE: _____ LENGTH: _____

SAND PACK: TSP: _____ TYPE: _____ LENGTH: _____

SURFACE COLLAR:

TYPE: _____ RADIUS: _____ THICKNESS CENTER: _____ THICKNESS EDGE: _____

CENTRALIZER DEPTHS

DEPTH 1: _____ DEPTH 2: _____ DEPTH 3: _____ DEPTH 4: _____

COMMENTS:

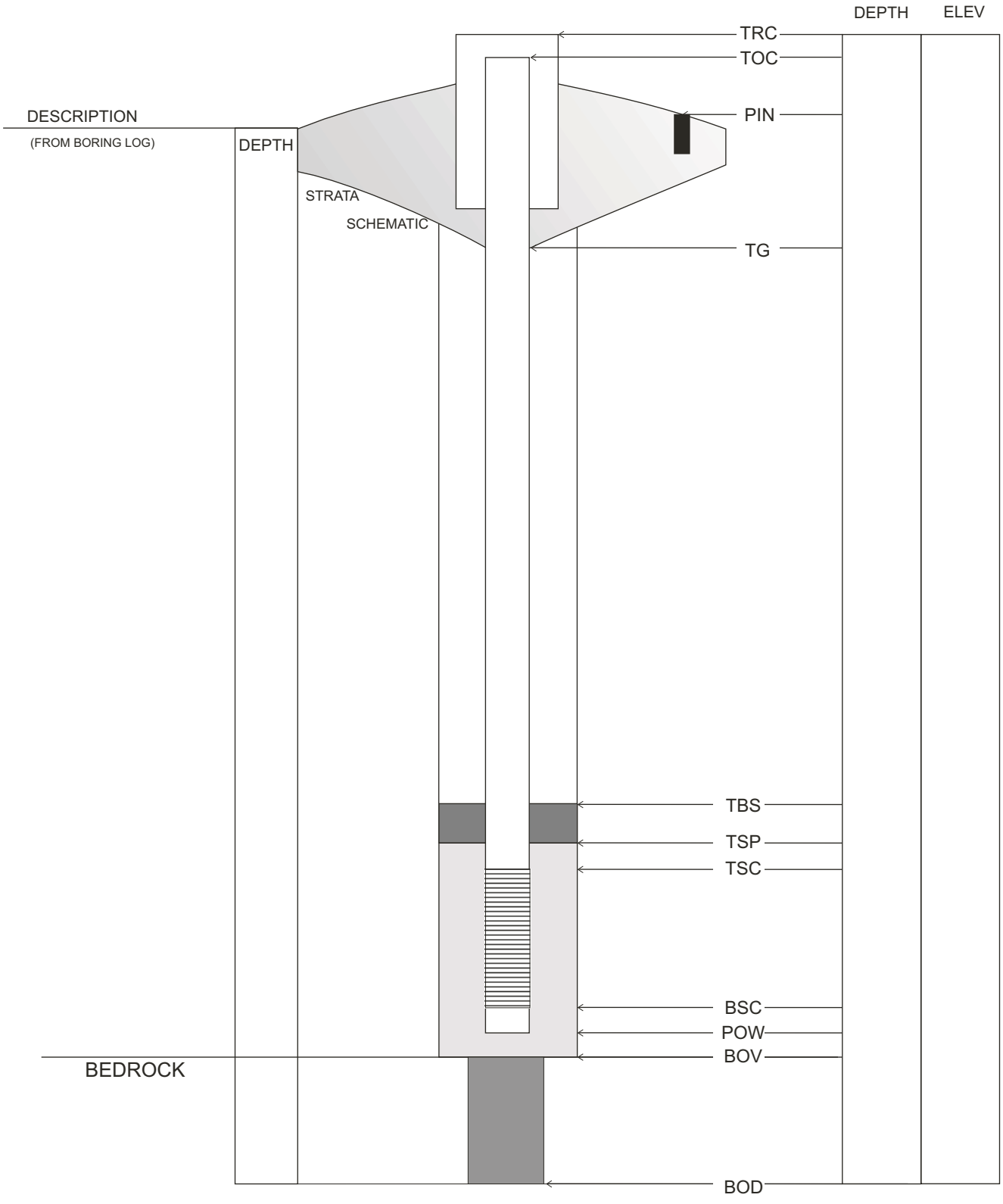
* ALL DEPTH MEASUREMENTS REFERENCED TO GROUND SURFACE

SEE PAGE 2 FOR SCHEMATIC

OVERBURDEN MONITORING WELL
PROTECTIVE RISER INSTALLATION DETAIL

PARSONS	CLIENT:	WELL #:_____

DATE:_____



BEDROCK MONITORING WELL
COMPLETION REPORT & INSTALLATION DETAIL
PROTECTIVE RISER COMPLETION

PARSONS | **CLIENT: USACOE** | **WELL #: MW**

PROJECT: _____ PROJECT NO: _____

SWMU # (AREA): _____ INSPECTOR: _____

SOP NO.: _____ CHECKED BY: _____

DRILLING CONTRACTOR: _____ POW DEPTH (ft) : _____

DRILLER: _____ INSTALLATION STARTED: _____

DRILLING COMPLETED: _____ INSTALLATION COMPLETED: _____

BORING DEPTH: _____ SURFACE COMPLETION DATE: _____

DRILLING METHOD(S): _____ COMPLETION CONTRACTOR/CREW: _____

BORING DIAMETER(S): _____ BEDROCK CONFIRMED (Y/N?) _____

PROTECTIVE SURFACE CASING

DIAMETER (ft): _____ LENGTH (ft): _____

RISER

TYPE: _____ TR (ft): _____

DIAMETER(in): _____ LENGTH (ft): _____

SURFACE COLLAR

TYPE: _____ RADIUS (ft): _____

THICKNESS OF CENTER (ft): _____ THICKNESS OF EDGE (in) _____

SCREEN

TYPE: _____ TSC (ft): _____

DIAMETER (in): _____ SLOT SIZE: _____ LENGTH (ft): _____

OUTER CASING

TYPE: _____ TC (ft): _____

DIAMETER (in): _____ POC (ft): _____ LENGTH (ft): _____

POINT OF WELL (SILT SUMP)

TYPE: _____ BSC (ft): _____ POW(ft): _____

GROUT

TYPE: _____ TG (ft): _____ LENGTH (ft): _____

SEAL

TYPE: _____ TBS (ft): _____ LENGTH (ft): _____

SAND PACK

FINE SAND TYPE: _____ TSP (ft): _____ LENGTH (ft): _____

COARSE SAND TYPE: _____ TSP (ft): _____ LENGTH (ft): _____

ACRONYMS

TR	Top of Riser	BSC	Bottom of Screen	TG	Top of Grout
TSC	Top of Screen	POW	Point of Well	TBS	Top of Bentonite Seal
BGD	Background	TSP	Top of Sand Pack		

COMMENTS:

* ALL DEPTH MEASUREMENTS REFERENCED TO GROUND SURFACE

WELL DEVELOPMENT REPORT

PARSONS	CLIENT : USACOE	WELL #: MW

PROGRAM TYPE :	CREW INITIALS	START DATE	END DATE
SWMU # (AREA) :			
PROJECT NO. (JOB #):			

DRILLING DATE:	MONITORING	BEFORE DEVELOPMENT		AFTER DEVELOPMENT	
INSTALLATION DATE:	INSTRUMENT	OVM	RAD	OVM	RAD
SOP REFERENCE NO. & REV. NO. :	READING				
PUMP EQUIPMENT:	UNITS (ppm or cps)				

WELL TYPE (circle one)	BEDROCK	OVERBURDEN	MEASURED WATER DEPTH (feet from TOC):
WELL INNER RISER DIAMETER (inches)	2	2	MEASURED POW DEPTH (feet from TOC):
WELL DIAMETER FACTOR (gal/ft)	0.163	0.163	WATER COLUMN (feet) :
BORING DIAMETER (inches)	3.80	8.5	INSTALLED WATER DEPTH (feet from TOC):
BORING DIAMETER FACTOR (gal/ft)	0.5894	2.955	INSTALLED POW DEPTH (feet from TOC):

1. STANDING VOLUME INSIDE WELL = WATER COLUMN X WELL DIAMETER FACTOR = _____ GAL. = A

2. STANDING WATER IN ANNULAR SPACE =
WATER COLUMN BELOW SEAL(ft) X (BORING DIAMETER FACTOR - WELL DIAMETER FACTOR) X 0.3 = _____ GAL. = B

3. SINGLE STANDING WATER VOLUME = A + B _____ GAL. = C

4. MINIMUM VOLUME TO BE REMOVED = 3 X C _____ GALS.

DATE	ACTIVITY	START TIME (military)	END TIME (military)	GALLONS REMOVED PER TIME PERIOD	pH	CONDUCTIVITY (umhos/cm)	TEMPERATURE (degrees C)	TURBIDITY (NTUs)
	TOTALS/FINAL							

COMMENTS:

INVESTIGATION DERIVED WASTE (IDW) :

DATE			
GALLONS OF WASTE WATER			
DRUM NO. & LOCATION			

GROUNDWATER ELEVATION REPORT

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

MONITORING EQUIPMENT:					WATER LEVEL INDICATOR:		COMMENTS:
INSTRUMENT	DECTECTOR	BGD	TIME	REMARKS	INSTRUMENT	CORRECTION FACTOR	

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)	(FROM) DIRECTION (0 - 360)	GROUND / SITE SURFACE CONDITIONS	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)]

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/ DATE
					COUNT/ VOLUME	TYPE			
1	VOC -CLP(Low Level) 8260B		4 deg. C	HCL	3/ 40 ml	VOA			
2	SVOC 8270C		4 deg. C		1 x 1L	Am G			
3	PESTICIDES 8081		4 deg. C		1 x 1L	Am G			
4	PCBs 8082				1 x 1L				
5	METALS 6010 & 7###		4 deg. C	HNO3	1 x 500 mL	HDPE			
6	CYANIDE 9012		4 deg. C	NaOH	1 x 500 mL	HDPE			
7	Total Pet Hydrocarbon		4 deg. C	HCl	1 x 1L	Am G			

COMMENTS: (QA/QC?)

IDW INFORMATION:

SAMPLING RECORD - SURFACE SOIL/SEDIMENT

PARSONS | **CLIENT: USACOE** | **INSPECTOR :** | **DATE:**

PROJECT:

Plume Area:

SOIL TYPE

SURFACE SOIL SEDIMENT

COMMENTS:

MONITORING

INSTRUMENT	DETECTOR	READING

SAMPLE INFORMATION					SOIL INFORMATION					
LOCATION	SAMPLE NUMBER	SAMPLE DEPTH (in) TOP	SAMPLE DEPTH (in) BOTTOM	TIME (military)	GRAB or COMPOSITE SAMPLE	SAMPLE DESCRIPTION (Burmister method)	USCS Classification	VOC Screen (PPM)	QC Split (yes or no)	Other Notes

SAMPLING RECORD - SOIL

Seneca Army Depot Activity	PARSONS	DATE:

CONSULTANT: _____ INSPECTOR: _____
PROJECT: _____ LABORATORY: _____
LOCATION: SAMPLING STAFF: _____
CHAIN OF CUSTODY #: _____

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

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

LOC ID	SAMPLE #	DEPTH RANGE	DEPTH TIME	TYPE GRAB/COMP	COLOR	GRAIN SIZE	USCS CLASS	FOREIGN MAT. (Y/N)	SAMPLE DEVICE	CONTAINER SIZE/TYPE	MON. VOC/RAD	QC SPL (Y/N)

Note: Cleaning Procedure according to SOP.

SAMPLING RECORD - SURFACE WATER

SENECA army DEPOT ACTIVITY | **PARSONS** | **DATE:**

CONSULTANT: _____
PROJECT: _____
LOCATION: _____

INSPECTOR: _____
LABORATORY: _____
LAB. STAFF: _____
CHAIN OF CUSTODY #: _____

WEATHER / FIELD CONDITIONS CHECKLIST (RECORD MAJOR CHANGES)

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

LOC ID	SAMPLE #	DFS	DEPTH	TIME	TEMP	MON. VOC	MON. CLR	TURBIDITY (NTU)	pH	SPEC COND	D.O.	SAMPLING DEVICE	CONT TYPE / SIZE	QC SPL (Y/N)

DFS-DISTANCE FROM SHORE (FEET)
IDENTIFY UNITS FOR ALL MEASURMENTS
CLEANING PROCEDURES ACCORDING TO SOP

SAMPLING RECORD - SURFACE WATER

SENECA ARMY DEPOT ACTIVITY				PARSONS			WELL #: MW		
SAMPLING ORDER		TAL/ TCL	PRESERV.	BOTTLES		SAMPLE NO.	DATE/TIME		CHECKED BY/
				COUNT/ VOLUME	TYPE				DATE
1	VOA/CLP			3/ 40 ml	G. vial				
1A	VOA/524.2			3/ 40 ml	G. vial				
2	SVOC			2/ 1 L	G. Amber				
3	HERB			2/ 1 L	G. Amber				
4	PEST/ PCB			2/ 1 L	G. Amber				
5	METALS + Sn			1/ 1 L	P				
6	CN			1/ 1 L	P				
7	TPH			2/ 1 L	G				
8	Hardness								
9	TDS								
10	COD								
11	SULFIDE			1/ 500 ml	G				
12	CATIONS K, Mn, Mg, Fe, Na			1/ 1 L	G. Amber				
13	ANIONS			1/ 1 L	P				
14	AMMONIA			1/ 1 L	P or G				
15	GROSS ALPHA/BETA			1/ 1 Gallon	P				

QA/QC

BOTTLE COUNTS ARE TRIPLED IF MS/ MSD SAMPLES ARE COLLECTED

QA/QC DUPLICATE SAMPLE COLLECTED? YES NO

Duplicate Sample Name: _____

MRD Sample Name: _____

QA/QC rinsate sample name: _____

MATRIX SPIKE sample collected? YES NO

INVESTIGATION DERIVED WASTE (IDW):

DATE:				
VOLUME:				
DRUM #, LOCATION:				

COMMENTS:

Appendix H

Reference Manuals for Field Measurement Instruments

48440-18

DR/820, DR/850, and DR/890

Portable Datalogging Colorimeter Instrument Manual

dd/dp 6/02/97 1ed
rev. 5, 8/99

TABLE OF CONTENTS

TABLE OF CONTENTS, continued

Before attempting to unpack, set up or operate this instrument, please read this entire manual. Pay particular attention to all warnings, cautions and notes. Failure to do so could result in serious injury to the operator or damage to the equipment.

To ensure the protection provided by this equipment is not impaired, this equipment MUST NOT be installed or used in any manner other than that which is specified in this manual.

Use of Hazard Information

If multiple hazards exist, the signal word corresponding to the greatest hazard shall be used.

DANGER

Indicates either a potentially or an imminently hazardous situation which, if not avoided, could result in either death or serious injury

CAUTION

Indicates a potentially hazardous situation that may result in minor or moderate injury

NOTE

Information that requires special emphasis

Precautionary Labels

Please pay particular attention to labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed.

The DR/800 Series Colorimeters are Class 1 LED products. A Class 1 LED product has insufficient energy to be considered an eye hazard.

⚠ This symbol, if noted on the instrument, references the Instruction Manual for operational and/or safety information.

⚠ **Section *2.1 Battery Installation***

⚠ ***SECTION 4 CREATING USER-ENTERED PROGRAMS***

SPECIFICATIONS

Specifications subject to change without notice.

Wavelength Range(s):

- Model DR/890: 420, 520, 560, 610 nm
- Model DR/850: 520, 610 nm
- Model DR/820: 520 nm

Wavelength Accuracy: ±1 nm

Wavelength Selection: Automatic

Photometric Linearity: ±0.002 A (0-1 A)

Photometric Reproducibility: ±0.005 A (0-1 A)

Photometric Accuracy: ±0.005 A @1.0 ABS Nominal

Source Lamp: Light Emitting Diode (LED)

Detector: Silicon Photodiode

Data Readout: 4-digit LCD, 1.5-cm Character Height

Readout Modes: % Transmittance, Absorbance, Concentration

External Outputs: IR (Infrared to RS-232 Serial using the Data Transfer Adapter)

Battery Power: (4) AA alkaline cells

Instrument Dimensions: 23.6 x 8.7 x 4.7 cm (9.3 x 3.4 x 1.9 inches)

Instrument Weight: 470 g (1 lb.)

Photometric Range: 0-2 A

Stray Light: <1.0% at 400 nm

Battery Life: 6 months (typical)

Temperature Range:
Operating Range: 0 to 50 °C (32 to 122 °F)
Storage Range: -40 to 60 °C (- 40 to 140 °F)

Humidity: 90% at 50 °C

Environmental: Designed to meet IP67 Standard; dustproof and waterproof

OPERATION

DANGER

Handling chemical samples, standards, and reagents can be dangerous. Review the necessary Material Safety Data Sheets and become familiar with all safety procedures before handling any chemicals.

DANGER

La manipulation des échantillons chimiques, étalons et réactifs peut être dangereuse. Lire les Fiches de Données de Sécurité des Produits (FDSP) et se familiariser avec toutes les procédures de sécurité avant de manipuler tous les produits chimiques.

PELIGRO

La manipulación de muestras químicas, estándares y reactivos puede ser peligrosa. Revise las fichas de seguridad de materiales y familiarícese con los procedimientos de seguridad antes de manipular productos químicos.

GEFAHR

Das Arbeiten mit chemischen Proben, Standards und Reagenzien ist mit Gefahren verbunden. Es wird dem Benutzer dieser Produkte empfohlen, sich vor der Arbeit mit sicheren Verfahrensweisen und dem richtigen Gebrauch der Chemikalien vertraut zu machen und alle entsprechenden Materialsicherheitsdatenblätter aufmerksam zu lesen.

PERIGO

A manipulação de amostras, padrões e reagentes químicos pode ser perigosa. Reveja a folha dos dados de segurança do material e familiarize-se com todos os procedimentos de segurança antes de manipular quaisquer produtos químicos.

SECTION 1 GENERAL DESCRIPTION

1.1 Instrument Description

The Hach DR/800 Series Colorimeter shown in *Figure 1* is a microprocessor-controlled, LED-sourced filter photometer suitable for colorimetric testing in the laboratory or the field. The instrument is precalibrated for common colorimetric measurements and includes convenient calibration capability for user-entered and future Hach methods. Instrument features include:

- Test results are displayed in concentration, absorbance, or percent transmittance.
- Automatic wavelength selection and ranging in the preprogrammed parameters.
- Data storage and recall for datalogging in the field or laboratory.
- Conversion of results to alternate forms for many parameters (i.e., PO_4, P_2O_5, P).
- Reagent Blank Correction and Standard Adjust features may be used to compensate for lot-to-lot variations in reagents.
- Icon prompts displayed during testing.
- A built-in timer to monitor specific reaction times called for in the test procedures. Appropriate times are programmed into the calibration data for specific tests. The timer also can be used manually by the operator independent of the stored methods.
- IR output for RS232 interface capability allows an external printer or computer to interface with the colorimeter.
- Entry of user-entered methods or new Hach methods.
- Error signals for procedural or instrument troubleshooting.

The colorimeter operates on battery power. The instrument holds four AA-size alkaline dry cells (batteries supplied) that power the instrument for at least six (6) months. Optional rechargeable alkaline batteries are also available. The charger and optional rechargeable batteries must be purchased separately.

Figure 1 DR/800 Series Colorimeter Standard Package*

1.2 Unpacking the Instrument

Remove the instrument and accessories from the shipping container and inspect each item for any damage that may have occurred during shipping. Verify that all items listed on the packing slip are included. If any items are missing or damaged, please contact Hach Customer Service, Loveland, Colorado for instructions.

* Carrying Case may be ordered separately.

Hach's toll-free number for customers within the United States is 800-227-4224. For customers outside the United States, contact the Hach office or distributor serving you.

1.2.1 Standard Accessories

- Sample Cells — (2) round, with 10-mL, 20-mL, and 25-mL marks
- COD/TNT Adapter for use with 16-mm vials used in COD and Test 'N Tube methods.
- Batteries — (4) AA alkaline
- Documentation package — includes Instrument Manual and Procedures Manual.

In addition to these standard accessories, several other optional accessories are available from Hach Company (refer to *Replacement Parts and Accessories*).

1.2.2 Optional Accessories

- Immunoassay Tube Adapter
- Rechargeable Alkaline Batteries
- External Alkaline Battery Charger
- Data Transfer Adapter (for RS232 interface)
- HachLink™ Software
- Portable Printer
- Instrument case
- DR/Check™ ABS Standard

1.3 Description of the Keypad

Figure 2 shows the colorimeter's keypad. The description and function of each individual key is given in *Table 1*.

Figure 2 Keypad

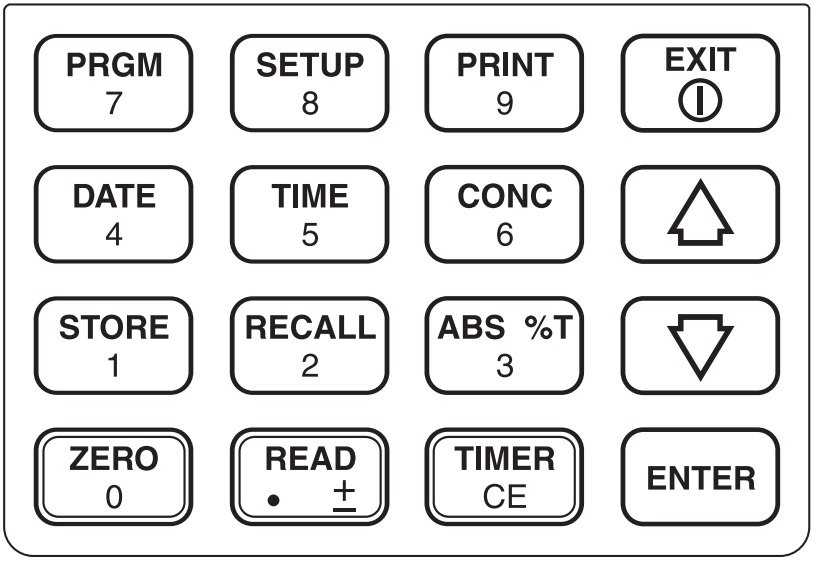


Table 1 Keys and Descriptions

KEY	DESCRIPTION
PRGM 7	Allows the user to select a program. Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen.
SETUP 8	Accesses the SETUP menu (the SETUP icon illuminates in the upper left-hand corner of the display screen). The setup menu provides access to options such as reagent blank, standard adjust, user-entered programs, and instrument configurations. Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen.
PRINT 9	Prints currently displayed data. In the RECALL menu, prints recalled data. Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen.
EXIT ⏻	Use this key to turn the instrument on and, when the instrument is on and the EXIT icon is not illuminated, press this key to turn the instrument off. When the EXIT icon is illuminated at the base of the display screen, the **EXIT** key cancels the current entry or selection.

Table 1 Keys and Descriptions (Continued)

KEY	DESCRIPTION
DATE 4	Displays the current date. In the **RECALL** menu, displays the date the recalled sample was stored. Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen. Used to set the current date from the **SETUP** menu.
TIME 5	Displays the current time. In the **RECALL** menu, displays the time the recalled sample was stored. Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen. Used to set the current time from the **SETUP** menu.
CONC 6	When performing an analysis, this key displays the concentration value of the reading. Used as a toggle key to access alternate chemical forms, if available. Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen.
△	Scrolls up through selected menus or stored data.
STORE 1	When performing an analysis, this key allows the user to store a current reading in one of 99 sample locations. The user can store the reading as numbers 1-99 by pressing **ENTER**. Use the up and down arrow keys to find unused storage numbers or use numeric keys to enter a sample number. Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen.
RECALL 2	Begins the retrieval of stored sample readings (**RECALL** icon illuminates in the upper-left portion of the screen). Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen.
ABS %T 3	Toggles between displaying Absorbance and % Transmittance. Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen.
▽	Scrolls down through selected menus or stored data.
ZERO 0	Zeros the instrument on the current sample blank. Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen.
READ • ±	When the **READ** icon is illuminated at the base of the display screen, this key reads and displays the sample concentration. Also used as a numeric key function when the "#" icon is illuminated at the base of the display screen; the first press is a decimal, the second press toggles the value sign.
TIMER CE	If using a Hach-stored program, the **TIMER** key automatically sets the appropriate reaction time. If not in a Hach-stored program, the **TIMER** key allows the user to set a timer. When the "#" icon is illuminated at the base of the display screen**, CE** clears the most recent level of action (deletes the whole entry, not just the last number).
ENTER	Within a menu, selects the displayed menu item. During numeric entry, accepts the displayed value.

1.4 Display Screen in Function and Numeric Modes

The main display operates in two modes: function mode and numeric mode. The user does not select the mode, changeover is automatic depending on the options selected, where the user is in an analysis, and what information the instrument needs from the user.

The main display shows action icons (**ZERO** and **READ**) displayed below the horizontal line. This shows there are two available options to select (zero the instrument, or take a reading) at this point in the analysis.

The numeric mode is signified by the "#" icon illuminated below the horizontal line. In the numeric mode, some function keys act as numeric entry keys (corresponding to the number on the key).

1.5 Icon and Display Screen

Figure 3 shows the icons displayed by the DR/800 Series Colorimeters. *Table 2* provides a brief description of each display element.

Figure 3 Icon and Main Display Screen

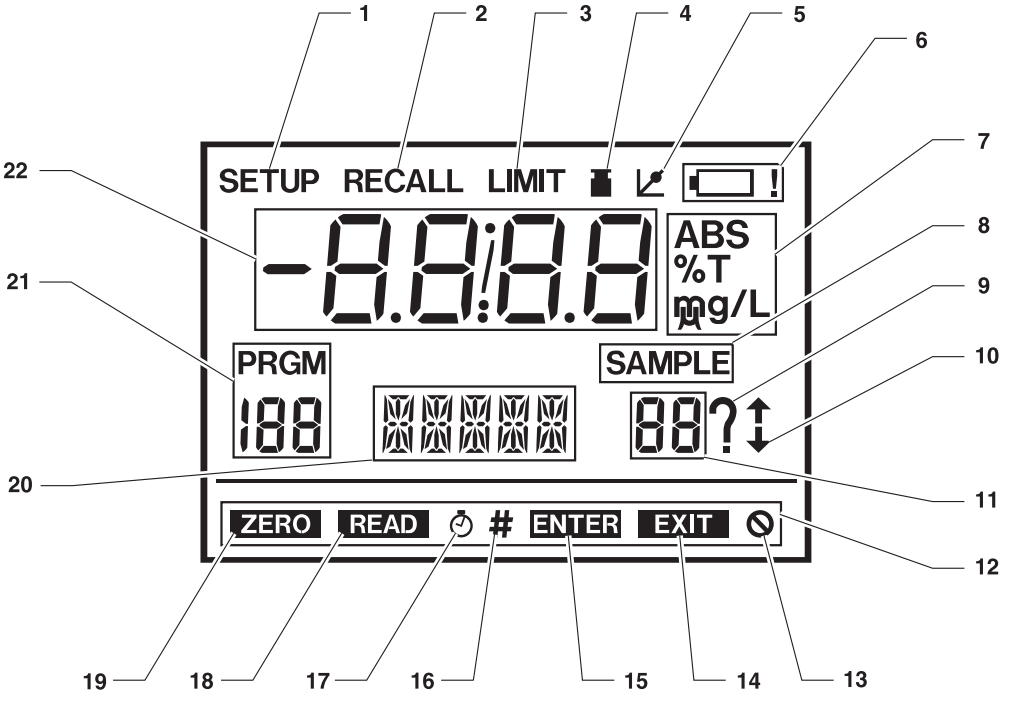


Table 2 Main Display Screen Icons

ITEM NO.	DEFINITION
1	Indicates the user is in the **SETUP** menu.
2	Indicates the user is in the **RECALL** menu.
3	The sample concentration exceeds the limits of the selected program.
4	Sample cell icon. Indicates a reagent blank adjustment is in use for the current program.
5	Standard adjust icon. Indicates a standard adjust is in use for the current program.
6	Indicates a low battery condition - replace the batteries as soon as possible.
7	These three icons follow the sample reading and represent either absorbance, percent transmittance, milligrams per liter, micrograms per liter, or grams per liter.
8	Illuminates whenever the numbers in the main display (22) or the sample display (11) refer to a sample number.
9	Indicates the instrument is waiting for information from the user.
10	Depending on the arrow(s) illuminated, these icons indicate the available scroll direction (using the **ARROW** keys) for accessing options.
11	In the RECALL menu or when storing data, these digits show the selected sample number.
12	Most icons displayed in this area are action icons. Action icons tell the user what actions are acceptable options during an analysis.
13	Indicates an invalid key press was made. This icon flashes briefly accompanied by a short beep.
14	Exit action icon - (when illuminated) tells the user that pressing the **EXIT** key to exit the current level of action is an acceptable option.
15	Enter action icon - (when illuminated) tells the user that pressing the **ENTER** key to confirm an action is an acceptable option.
16	Numeric entry action icon - (when illuminated) tells the user the numeric key pad is active.
17	Timer action icon - (when illuminated) tells the user that the instrument is presently running a timer. This icon will flash while the timer is counting.
18	Read action icon - (when illuminated) tells the user that pressing the **READ** key to read the sample cell is an acceptable option.
19	Zero action icon - (when illuminated) tells the user that pressing the **ZERO** key to zero the instrument on a sample cell is an acceptable option.
20	Depending on the currently active menu, the series of alphabetical letters displayed here gives information on a current reading, a stored reading, indicates the options available within a menu, or prompts the user for the next action.
21	Shows the active program number, (either a user-entered (101-110) or Hach-stored program (1-100). The program number is displayed immediately below the PRGM icon.
22	Depending on the currently active menu, the numbers displayed here represent either the sample reading, the clock timer, or the numeric characters entered by the user.

SECTION 2 INSTRUMENT SETUP

2.1 ⚠ Battery Installation

Power is supplied by four AA-sized alkaline batteries. Typically, a set of batteries provides approximately six months of operation. The colorimeter lamp is an LED and is on only long enough for the measurement sequence to take place (approximately 2 seconds).

The instrument will automatically shut off if no keystrokes are made for 15 minutes when in normal mode and four hours when in user-entry mode.

Figure 4 provides an exploded view of the battery installation. When replacing discharged batteries, always replace the complete set of four.

Hach recommends using alkaline batteries in this instrument. **Do not use rechargeable Nickel Cadmium (NiCad) batteries.** If rechargeable batteries are desired, rechargeable alkaline batteries are available from Hach.

The battery compartment is accessible from the underside of the instrument. Make sure the sample cell compartment is empty. Lay the instrument upside down on a padded surface, and install batteries as follows:

Note: *For performance reasons, never remove the battery cover from this product except when servicing the batteries.*

1. Disconnect the Data Transfer Adapter (if connected) from the instrument.
2. Loosen the two battery compartment screws and remove the battery compartment door as shown in *Figure 4*.
3. Install four AA alkaline batteries in the battery holder as shown in *Figure 4*. Match the polarities of the batteries with the polarity markings in the battery compartment.
4. Replace the battery compartment cover, tighten the screws, and return the instrument to the upright position.

DANGER ⚠
Use of nickel-cadmium batteries under a fault condition creates a potential fire hazard.

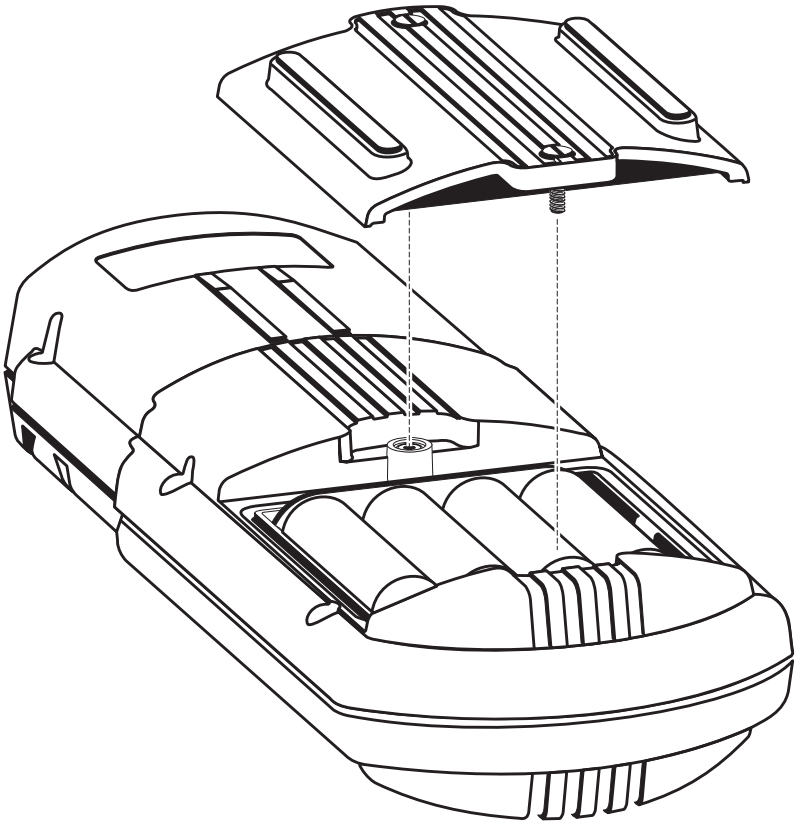
PELIGRO
La utilización de pilas de níquel-cadmio en condiciones de falla crea el riesgo de incendio.

PERIGRO
O uso de baterias de níquel-cádmio em condição de falha cria a possibilidade de incêndio.

DANGER
L'utilisation de batteries nickel-cadmium dans des conditions inappropriées crée un risque d'incendie.

GEFAHR
Unter einer Störungsbedingung stellt die Verwendung von Nickel-Kadmium-Batterien eine Feuergefahr dar.

Figure 4 Battery Installation



2.2 Turning the Instrument On

Once batteries are installed, turn the instrument on using the **EXIT/I/O**key (located on the top row, far-right column of the instrument keypad).

Press the key once to power-on the instrument. The display will show the software version number, then will default to the last used program number. The instrument is now ready for operation.

2.3 Setting the Date and Time

Setting the instrument's date and time allows sample readings to be stored and recalled with the proper date and time. Check the currently entered date or time by pressing the respective **DATE** or **TIME** key.

To set the date and time, continue with Section *2.3.1* or *2.3.2* below.

2.3.1 Entering the Correct Date

Check the current date by pressing the **DATE** key. If the date is incorrect, follow the procedure below to change it.

Enter the correct year, then the correct month and day as follows:

1. Press the **I/O** key to turn the instrument on.
2. Access the **SETUP** menu by pressing the **SETUP** key on the keypad. (The down **ARROW** icon on the right side of the display is shown.)
3. Press the down **ARROW** key until **DATE** is displayed.
4. Press the **ENTER** key to select the date option.
5. Four horizontal lines (showing available spaces for numeric entry) and **YEAR ?** will appear on the display. Enter the digits corresponding to the correct year using the numeric keypad. For instance, if the year is 1997, press **1 9 9 7** then the **ENTER** key.

   If an incorrect number is entered, press the **CE** key and re-enter the information.

Next, the instrument prompts for the month and day.

6. Enter the correct month and day using the numeric keypad. The month must be entered first, followed by the day. If an incorrect number is entered, press the **CE** key and re-enter the information.

Note: *When entering a one digit month or day, always press the* **ZERO** *key before the digit. For example: If the month and day to be entered is March 4, press* **0 3 0 4** *on the numeric keypad, then press the* **ENTER** *key to accept.*

7. Press the **ENTER** key to accept the new information. Press the **EXIT** key to return to the main menu

2.3.2 Entering the Correct Time

1. Press the **I/O** key to turn the instrument on.
2. Access the **SETUP** menu by pressing the **SETUP** key on the keypad. (The down **ARROW** icon on the right side of the display is shown.)
3. Press the down **ARROW** key until **TIME** is displayed.
4. Press the **ENTER** key to select the time option.
5. Enter the time in 24-hour (military) notation using the numeric keypad then press the **ENTER** key to accept the entry. For example, 9:00 a.m. is entered as **0 9 0 0 ENTER** and 2:00 p.m. is entered as **1 4 0 0 ENTER**. If an incorrect number is entered, press the **CE** key and re-enter the information.
6. The display returns to the setup menu. Press the **EXIT** key to return to the main menu.

2.4 Sample Cell Insertion

Wipe the sample cell with a lint-free cloth or tissue and insert the cellinto the sample cell compartment with the diamond-shaped marker toward the keypad.

Figure 5 Placing Samples in the Sample Cell Compartment

2.5 Sample Cell Adapter Installation

When installing an adapter into the sample cell compartment, insert the adapter into the cell compartment and rotate until it drops into the alignment slots. Finish installation by gently pushing down on the adapter until it snaps into position.

Place the appropriate vial or sample cell into the adapter - the vial or sample cell should fit well into the adapter. If it does not, double-check that the correct sample container (vial or sample cell) is being used and that the adapter is installed correctly. For a list of available adapters, refer to *Replacement Parts and Accessories*.

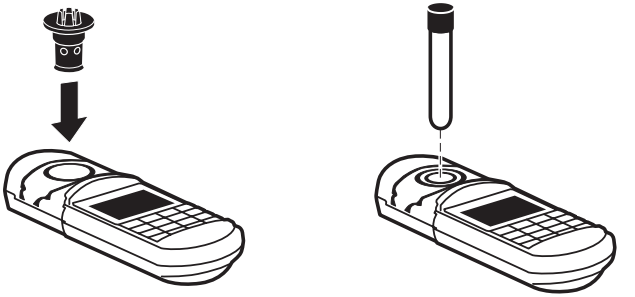
2.5.1 Using the 16-mm COD/Test ‘N Tube Vial Adapter

The methods for chemical oxygen demand (COD) and Test ‘N Tube (TNT) determinations in the colorimeter procedures manual use 16-mm vials as the sample cell for the colorimetric measurement. This adapter also holds a standard 16-mm test tube.

Place the COD/TNT Adapter in the instrument's sample cell compartment as instructed in Section *2.5*, above. Place the vial into the COD/TNT Adapter (see *Figure 6*).

Always place the instrument cap over the adapter when measuring in bright sunlight (see *Figure 8*).

Figure 6 Installing the COD/TNT Adapter



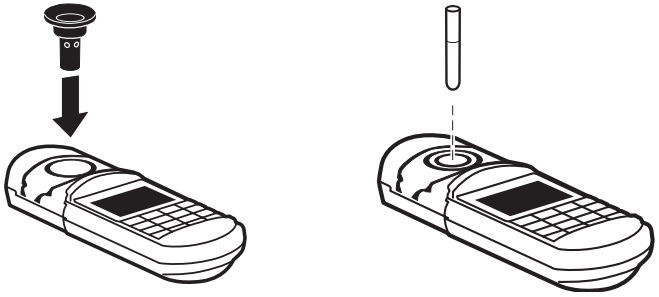
2.5.2 Using the Immunoassay Tube Adapter

Immunoassay methods develop and read the color in special antibody coated tubes. The instrument can read immunoassay results with the aid of the adapter.

Place the Immunoassay Tube Adapter in the instrument's sample cell compartment as instructed in section *2.5*, above. Place the tube into the adapter after it is properly installed in the cell compartment (see *Figure 7*).

Always place the instrument cap over the adapter when measuring in bright sunlight (see *Figure 8*).

Figure 7 Installing the Immunoassay Tube Adapter

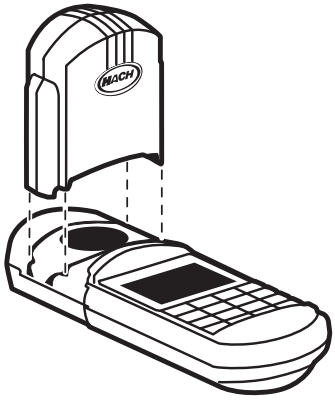


2.6 Using the Instrument Cap as A Light Shield

The colorimeter cap is removable (by sliding it away from the keypad) to expose the sample cell compartment. The instrument cap also functions as a light shield. Samples may be analyzed without the light shield in place but using the light shield provides a good seal against stray light and contributes to more accurate results. Use of the light shield is especially important when performing an analysis in bright light or direct sunlight.

To use the instrument cover as a light shield during measurements, place the cap over the sample cell, into the grooved marks on the instrument. See *Figure 8*.

Figure 8 Installing the Light Shield



SECTION 3 INSTRUMENT OPERATION

3.1 Menus

The instrument has two important menus which allow access to different options. The menus are:

- Setup Menu
- Recall Menu

Once the user has selected the desired menu, the arrow icons illuminate on the main display screen. The arrows indicate that additional options are available in this menu. Press the up or down **ARROW** key (whichever arrow is illuminated) to scroll until the desired option is displayed. A press of the **ENTER** key at this time selects the displayed option.

When choosing options from menus, the up or down **ARROW** keys, **ENTER,** and **EXIT** keys help navigate between menus and within menu options. Use the **ENTER** key to select a menu option. Press **ENTER** again to accept a new setting. Pressing the **EXIT** key leaves a menu or leaves the displayed item unchanged.

3.1.1 Setup Menu

Enter the **Setup Menu** from the **Main Menu** by pressing the **SETUP** key.

Refer to the arrow icons on the main display for an indication of which key (up **ARROW** or down **ARROW**) to press on the keypad - the direction of the illuminated icon on the main display indicates the available scrolling direction. The up and down **ARROW** (scroll) keys move from one menu option to the next.

Example: when the down arrow icon is illuminated on the display screen, the user only has the option to press the down **ARROW** key to move from one option to the next. Once the user scrolls down, the down arrow icon changes to a dual-direction arrow icon (up and down arrows) until the user reaches the last menu option available in that menu.

The following instrument operating features are available in the **Setup** menu

- **BLANK** - Used to compensate for color contributed by the reagents in a reagent blank. Readjust for each new lot of

reagents. Press the **ENTER** key to activate this option. The default setting is off. Chemistries which zero on a reagent blank do not have this option available.

- **STD** - Standard Adjust option allows for entry of the value of the prepared standard. Press the **ENTER** key to activate this option. The default setting is off.

Note: *The BLANK and/or STD options may or may not be available, depending on the selected Hach program; the BLANK and STD options are always available for user-entered programs.*

- **PRINT (ALL)** - Prints all stored data to a printer or downloads it to a personal computer. The information printed includes: sample concentration reading, date of sample, time of sample, units, sample number, program number, Absorbance, and %T. Refer to Section *SECTION 6* for detailed printing information and to *Figure 11* for an example of a printout. Press the **ENTER** key to activate this option.

- **USER** - Allows access to the User-Entered Program Menu. Press the **ENTER** key to activate this option. See *CREATING USER-ENTERED PROGRAMS* on page *35* for more information.

- **DATE** - Allows the user to set the date. Press the **ENTER** key to activate this option. See Section *2.3.1* for more information.

- **TIME** - Allows the user to set the time. Press the **ENTER** key to activate this option. See Section *2.3.2* for more information.

- **ERASE (ALL)** - This clears all the data previously stored in memory. Press the **ENTER** key to activate this option.

3.1.2 Recall Menu

The recall menu allows access to stored data. Complete information and instructions for the use of this menu is presented in *SECTION 5 DATA RECALL AND STORAGE*.

3.2 Performing an Analysis

The procedures manual provides illustrated, step-by-step procedures for performing all the factory-entered methods. This instrument manual has supplemental information on how the instrument performs the necessary functions, and how to use the special operating features. Once you are familiar with the

instrument, the instructions in the procedure manual should be sufficient to analyze your samples.

Colorimetric testing with preprogrammed calibrations can be divided into four general phases:

1. Colorimeter setup
2. Sample preparation
3. Zeroing the instrument
4. Measuring the prepared sample

The following sections describe each phase in detail.

3.2.1 Colorimeter Setup for Sample Analysis

The colorimeter setup using a Hach program begins by selecting the desired program number. Programs numbers can be found in the individual procedure. (See the DR/800 Series Procedures manual supplied with your instrument.) Prompting icons will appear to indicate which keys are acceptable for the user to select. After turning the instrument on, the main display shows information from the last program used before the instrument was turned off.

If a different program is desired, press the **PRGM** key and enter the desired program number with the numeric keypad. The instrument will recall that program.

If the number selected is not valid, an error signal sounds and the display momentarily flashes an error icon. The display returns to the prompt for the program number. Re-enter the correct program number. Only select chemistries are available on the DR/820 and DR/850. See *APPENDIX A AVAILABLE PARAMETERS AND RANGES* on page *59*.

When the program number has been successfully entered, the display immediately prompts the user to zero the instrument by illuminating the **ZERO** action icon.

3.2.2 Sample Preparation

For colorimetric tests, sample preparation is next. The zero solution (or blank) and sample are usually prepared at this time. Generally, sample preparation consists of adding the contents of a premeasured reagent pillow to a small volume of sample. Follow the instructions in the procedure specific to your analysis.

It is important to observe the waiting period specified in the test procedure to ensure the color from the reaction of the reagent(s) with the target analyte develops fully. Many procedures also give a maximum time limit after which the color may begin to fade.

The instrument has method-specific timers for color development times pre-programmed into the software. The user is notified when the time has elapsed by a series of short beeps.

3.2.3 Zeroing the Colorimeter

The instrument must be zeroed for each test or series of tests to establish a zero reference for the measurement. This is done by placing a solution recognized as the blank solution in the cell holder, covering the sample with the instrument cap, and pressing the **ZERO** key. The next prompt will display zeros and illuminate the **READ** action icon. The instrument is now ready to take the first sample reading.

Note: *Once the zero reference point has been established, several samples can be measured consecutively by placing each of them into the cell holder and pressing the* **READ** *key. The instrument can be re-zeroed at any time by placing the zero solution (blank) into the instrument and pressing the* **ZERO** *key.*

3.2.4 Measuring the Prepared Sample

When ready to take the reading, place the prepared sample in the sample compartment. For best results orient the sample cell consistently for each measurement; see *Figure 5* on page *21*. Place the instrument cap (light shield) over the sample cell and press the **READ** key. After a brief pause, the results will be displayed.

Toggle between absorbance or percent transmittance values by successive presses of the **ABS %T** key. Press the **CONC** key to restore the concentration display. Successive presses of the **CONC** key toggles between alternate forms, if any. See Appendix A for currently available chemistries and their alternate forms.

3.2.5 Alternate Chemical Forms

Many Hach programs provide alternate chemical forms for the measured parameter. Press the **CONC** key to scroll through these alternate forms after the measurement is displayed. Each press of the **CONC** key takes the user to the next alternate chemical form. For example: In Program #1, mg/L Al may also be displayed as mg/L Al_2O_3. If alternate forms are not available, the instrument returns to its original form and reading.

3.2.6 Using the Timer

Many Hach test methods use one or more timers which are pre-programmed into the DR/800 Series Colorimeters. When the procedure instructs you to, press the **TIMER** key to display a timer interval. Press **ENTER** to start the timer count-down. Several beeps will sound at the end of a timer interval. If the method requires additional timers, the instrument will automatically display the next timer when the first timer elapses. Press **ENTER** to start the next timer.

To zero the instrument on the blank while the timer is running, press the **EXIT** key. The timer icon will continue to flash, indicating the timer is running. Press **ZERO**, or perform other available functions within the method (such as blank correction) while the timer is running. To return to the timer display, press the **TIMER** key.

3.2.6.1 Using the Timer in Manual Mode

The manual timer function allows the operator to use the timer independently from the method timer. Make sure the instrument is not in numeric entry mode and activate the timer with a press of the **TIMER** key. If using a pre-programmed timer, press the **TIMER** key again.

The "#" action icon is illuminated, to indicate that the numeric keypad is active. Enter the desired time using the numeric keypad. For example, to enter 2 minutes, press **2 0 0,** then **ENTER.** To enter 12 minutes, press **1 2 0 0,** then **ENTER**. The display will momentarily show the entered time, then the timer countdown will begin. The display will show the remaining time. At the end of the elapsed time, the instrument sounds five beeps.

3.2.6.2 Stopping the Timer

Stop the timer at any point in the countdown by pressing the **ENTER** key. If a pre-programmed timer was in use, the full timer period will be displayed. Press the **ENTER** key again to resume count down. If a manual timer was in use, the display for entering a new length of time will be shown, see Section *3.2.6.1 Using the Timer in Manual Mode*. Press **EXIT** to leave the timer mode.

3.3 Reagent Blank Correction

The Reagent Blank Correction can be used with some of the factory-entered methods. It 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; record the value for use in step *4.*.
3. Press **SETUP**, scroll to **BLANK** and press **ENTER**. The display will show **BLANK?**.
4. Enter the blank value obtained in step *2.*.
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 with the method) 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 attempt to use the Reagent Blank Adjust feature if the procedure uses a reagent blank for zeroing.

3.4 Adjusting the Standard Curve

The DR/890 Colorimeter has over 90 Hach Programs permanently installed in memory (other models have fewer programs). A program usually includes a preprogrammed 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 preprogrammed curve may not be convenient:

- Running tests where frequent calibration curve checks are required.
- 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 preprogrammed 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 preprogrammed 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. Enter the concentration of the standard used.

7. Press **ENTER**. The standard adjust icon 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 the error icon and the operation will not be allowed.*

To eliminate the standard curve adjustment:

1. Press the **PRGM** key.

2. Enter the stored-program number and press **ENTER**.

3. Press the **SETUP** key

4. Press an **ARROW** key to display **STD**.

Note: *On will be displayed if an adjustment is in use.*

5. Press **ENTER** twice.

3.5 Using a Programmed Method

Hach programmed and user-entered program options are available on this instrument. The Procedures Manual supplied at the time of purchase contains all currently available Hach-programmed methods. Up to ten user-programmed methods may be entered into the instrument. See *CREATING USER-ENTERED PROGRAMS* on page *35* for instructions for this function. *Table 3* shows the components of typical factory programmed test method.

Table 3

Step	Action/Keystroke	Display
1. Turn power on.	Press the **EXIT/I/O** key.	The instrument defaults to and displays the initial screen of the last program used. EXAMPLE: If program 20 was the last program in use, the instrument will automatically recall program 20.
2. Select the Program number to be used.	Press the **PGRM** key, then enter the program number and press the **ENTER** key.	After the **PGRM** key is pressed, a blinking cursor appears with a question mark. Enter the desired program number. Press **ENTER** to confirm this action and recall the desired program number.
3. As needed, set, then start the **TIMER**.	Press the **TIMER** key. In a Hach-stored program, the timer automatically defaults to the appropriate reaction time. Press **ENTER** to start the timer.	The entered or programmed reaction time will display and count down to zero.
4. ZERO the instrument using the sample blank.	Insert blank and press **ZERO.**	After the **ZERO** key is pressed, the instrument zeros on the sample cell.
5. Obtain reading in concentration, absorbance, or % transmittance.	Place the prepared sample into the cell holder. Press **READ**.	The instrument reads the sample and displays the results.

3.6 Quality Assurance

Verify the performance of your instrument in seconds using Hach DR/Check™ ABS Secondary Standards (Cat. No. 27639-00). These gel standards provide a measure of instrument absorbance for use anywhere, anytime, as often as laboratory quality assurance procedures suggest. The kit includes a blank and one standard each of a low-, mid-, and high range absorbance value from 0 to 2 ABS, for use with any DR/800 Series Colorimeter.

SECTION 4 CREATING USER-ENTERED PROGRAMS

DANGER
This instrument is not intended for use with flammable samples or those containing hydrocarbons.

PELIGRO
Este instrumento no está destinado para uso con muestras inflamables o que contengan hidrocarburos.

PERIGO
Este instrumento não é feito com o fim de ser empregado com amostras inflamáveis ou aquelas que contêm hidrocarbonetos.

DANGER
Cet instrument n'est pas conçu pour une utilisation avec des échantillons inflammables ou des échantillons contenant des hydrocarbures.

GEFAHR
Dieses Gerät darf nicht für Tests mit brennbaren Proben oder Proben, die Kohlenwasserstoffe enthalten, benutzt werden.

The DR/800 Series Colorimeters can store the calibration information needed to read prepared samples from up to five different user-entered programs. To create a new, user-entered program, you will need a blank and prepared standards or the correct absorbance readings for each standard. The prepared standards are made with standard solutions of the parameter (i.e. analyte). Up to twelve concentrations of standards, including a zero concentration standard, may be used.

The absorbances of the prepared standards must be different from one another. If the colorimeter detects a duplicate, it will beep and ignore the latest reading.

Using a previously entered method's program number erases all the previously entered information stored under that program number.

While creating a user-entered program, the colorimeter remains on for four hours following any keypress. If more than four hours pass between keypresses, the instrument will power down. All data which was entered, but not yet stored, will be deleted. The user-entered program must be re-entered from the beginning.

4.1 User-Entered Programs

The instrument allows storage of up to five user-entered programs (101-105) and up to 113 Hach programs.
A minimum of two data points are required for the instrument to recognize and accept a user-entered program.

- Program numbers 101 through 105 are reserved for storing user-entered programs.
- The maximum number of data points that can be entered for a method is 12. After the twelfth standard (1 through 12) is accepted, the instrument stores the method and will not accept any more data, but will allow the user to review the data already entered.

Before entering a calibration, determine the optimum wavelength, timing sequences (if any), and the workable range of the method.

4.2 Calibration Curves

Calibration curves may have positive or negative slopes, but they must be based on absorbance (% transmittance not allowed) and must pass through the origin that represents zero concentration.

It is important that the standards adequately describe the curve over the range of interest. Because this is largely dependent on the shape of the curve, it may be necessary to prepare a preliminary curve using extra data points to help select the appropriate standards.

If the curve is linear, only two concentration data points are needed. For example, standards with a zero absorbance and a standard with 1.000 absorbance are appropriate. If the curve is nonlinear, additional data points are needed to achieve good accuracy. Up to 12 data points can be entered for a single calibration curve.

4.3 User-entered Program Information for Bleaching Chemistries

Although the majority of colorimetric test procedures produce a higher absorbance (i.e. deeper color) as the concentration of the parameter being measured increases, some tests do the opposite. These bleaching chemistries (fluoride is one example) produce a lighter color at increasing concentrations. The zero concentration

standard is usually produced by combining deionized water with the reagents. Commonly this solution is used to zero the instrument as in Step *step 14* of *Section 4.4 Creating a New User-entered Program*.

Once the zero is entered, the prepared standards must be read from lightest to darkest. In the case of bleaching chemistries, the absorbance values reported by the colorimeter may be negative.

Even when your test produces a lower absorbance (lighter color) with increasing concentration, the prepared standards must be read by the colorimeter in the order of increasing absorbance (i.e., from colorless or the palest color, to the deepest color). The instrument will not accept standards read out of order.

4.4 Creating a New User-entered Program

Use the step-by-step instructions below to enter a new user-entered program into instrument memory. Terminate at any point (before the program is stored) by pressing the **EXIT** key until the display is blank. The colorimeter will not retain any of the entered data.

1. Press the **I/O** key to turn on the instrument.
2. Press the **SETUP** key. The display will show **SETUP** in the upper-left and the down-arrow icon in the lower-right. Available action functions are also shown.
3. Press the down **ARROW** key until **USER** is displayed.
4. Press the up **ARROW** key if the display goes past **USER**.
5. Press the **ENTER** key. Four horizontal lines (numeric entry display) will be displayed.
6. Select a program number from 101 through 105 by pressing the corresponding digit key. The number will appear in the display.

Note: *Press* **CE** *to correct errors.*

7. Press **ENTER**. A wavelength and **nm** will be displayed.

- If the wavelength is correct as displayed, skip to Step *step 8*.

- Some instrument models can use different wavelengths. If a different wavelength is preferred, proceed as follows:

a. Press **ENTER**. A flashing question mark will be displayed in the lower-right corner.

b. Press either **ARROW** key until the preferred wavelength is displayed.

c. Press **ENTER** to accept the displayed wavelength. The down-arrow icon will be displayed.

8. Press the down **ARROW** key to move to the **RES** (resolution) option. One to four zeros, a decimal point if needed, and the units of concentration may be modified here.

- If the displayed resolution and units are correct, skip to step *step 9*.

- If the displayed resolution or units are incorrect for your test, proceed with the following:

a. Press **ENTER**. A flashing question mark will be displayed.

b. Press either **ARROW** key until the preferred resolution and concentration units are displayed. The available options are:

0.000	0.00	0.0	0
0.000 µg/L	0.00 µg/L	0.0 µg/L	0 µg/L
0.000 mg/L	0.00 mg/L	0.0 mg/L	0 mg/L
0.000 g/L	0.00 g/L	0.0 g/L	0 g/L

c. Press **ENTER**. The question mark will disappear.

9. Press the down **ARROW** key to scroll to **STD**. **STD** and the number of the standard (i.e., 1 is shown for the first standard, 2 for the second, etc.) will be shown on the lower portion of the display.

10. Press **ENTER**. Four horizontal lines (denoting numeric entry) will be displayed.

11. Enter the standard's concentration, using the numeric entry keys (the # icon will be illuminated on the display).

Note: *Press the* **CE** *key to correct errors.*

12. Press the **ENTER** key. The concentration will be displayed.

Note: *A beep means that the concentration is a duplicate of a previous standard or the concentration is too high for the selected resolution. Repeat step step 11 with a different concentration and continue.*

13. Press the down **ARROW** key. **ABS** will be displayed followed by the number of the standard.

14. The colorimeter requires one zero be entered in this procedure; the **ZERO** action icon will appear in the lower portion of the display. Place a blank into the cell holder and press the **ZERO** key. Four horizontal lines will appear, then disappear, across the display. The **READ** action icon will appear in the lower portion of the display.

Note: *If necessary, the colorimeter can be re-zeroed. The most recently entered zero will be used for subsequent readings.*

15. Prepare the standards using the same reagents and procedure used to test samples.

16. Place the prepared standard into the cell holder.

17. Press the **READ** key. An absorbance value will be displayed.

Note: *Or, press the* **ENTER** *key to input an absorbance value or change the value read by the instrument. Use the numeric keys to enter the value then press the* **ENTER** *key.*

Note: *A beep indicates that the absorbance is a duplicate of a previously entered standard or that it falls between two previous standards. Repeat steps step 15 through 17. with the correct standard, or press the up* **ARROW** *key and repeat steps 9. through 17. with a correct, prepared standard and blank.*

18. Press the down **ARROW** key to advance to the next standard.

19. Repeat steps *step 9* through *step 18* for all remaining standards.

20. Press the **EXIT** key once. **STORE ?** will be displayed.

21. Press the **ENTER** key to store the new method in the instrument's memory.

4.5 Reviewing and Editing User-Entered Programs

Note: *When a user-entered program is edited and stored, all stored data associated with that program is erased.*

All method information previously stored by the operator can be reviewed and changed to add, delete, or modify data points. At any point during the editing function, the operator can terminate the procedure and exit by pressing the **EXIT** key. No changes to the program will occur.

Because the standards must be read in the order of increasing absorbancy, data points may not be inserted into the middle of an existing user-entered program.

To review and edit previously stored user-entered programs:

1. Press the **I/O** key to turn the instrument on.

2. Press the **SETUP** key.

3. Scroll to the **USER** option and press **ENTER**.

4. Enter the program number of the method to review or edit and press the **ENTER** key.

5. Scroll through the calibration information using the **ARROW** keys. To avoid making changes, press the **EXIT** key.

6. To edit the data shown on the display, press **ENTER**. Make necessary changes, then press the **ENTER** key to return to reviewing the data.

7. Press **EXIT** once. **STORE?** will be displayed.

8. Press **ENTER** to store the program.

4.6 Erasing User-entered Programs

Note: *When a user-entered program is erased, all stored data associated with that program is also erased.*

User-entered programs are automatically deleted when another user-entered program is entered and stored in the previously entered method's storage number (101-105). They also may be erased from the instrument memory as follows:

1. Press the **I/O** key to turn the instrument on.
2. Press the **SETUP** key.
3. Scroll to the **USER** option and press **ENTER**.
4. Enter the program number of the method to be erased and then press **ENTER**.
5. Scroll to the concentration data for STD 1 using the down **ARROW** key. Press **ENTER**.
6. Press **CE**. Press **ENTER**.
7. Press the **EXIT** key. **ERASE?** will be displayed.
8. Press the **ENTER** key to erase the method or the **EXIT** key to retain it in memory.

To store sample data, press **STORE** after the sample measurement is displayed. Data must be stored if you wish to recall it later for review, downloading or printing. The following information is stored for each sample:

- instrument model
- instrument serial number
- chemical form
- concentration
- units
- absorbance
- %T
- date
- time
- sample number
- program number

After the **STORE** key is pressed, a flashing question mark icon is shown in the lower-right portion of the display. In the center of the display, the next available storage number will appear. If this storage location is acceptable, press **ENTER** to accept it.

To select an empty storage number (between 1 and 99), use the **ARROW** keys to scroll to the desired number or enter the desired number with the numeric keys. Press **ENTER** to accept. The instrument will store the data, then revert back to the measurement display.

5.1 Recalling Data

To recall data stored in the colorimeter, press the **RECALL** key.

Use the **ARROW** keys to scroll through the stored data. Only data numbers containing stored sample reading data (of the available storage numbers 1-99) are displayed in the RECALL menu. For

example, if the user has stored data into storage numbers 6, 10 and 15, those are the only accessible numbers in the Recall option. Available storage numbers are not shown as a Recall option because no data has been stored for recall purposes.

When a number of sample readings have been stored and a specific reading must be recalled, proceed as follows:

1. Press the **I/O** key to turn the colorimeter on.
2. Press the **RECALL** key to access the **RECALL** Menu.
3. A beep indicates that no data is presently stored.
4. Press the number key(s) or either **ARROW** key on the keypad to scroll to the desired sample number.
5. Press the **ENTER** key. The stored reading will be displayed.
6. A beep indicates that no data is stored as that sample number.
7. While the stored reading is displayed, press the **DATE** key or the **TIME** key to display the date or time the reading was stored. Press the **CONC** key to display the concentration data.
8. If other stored data is desired, press either **ARROW** key until the data is displayed.
9. Press **EXIT** to terminate data recall.

5.2 Erasing All Stored Data

Stored data can be erased and the memory of the instrument cleared using the procedure that follows.

1. Press the **I/O** key to turn the colorimeter on.
2. Press the **SETUP** key to enter the **SETUP** menu.
3. Scroll using the down **ARROW** key until **ERASE** and **ALL** appears on the display.
4. Press the **ENTER** key to confirm this selection with the instrument.

A flashing question mark icon will appear in the lower-right portion of the screen as an additional step to avoid erasing all data if the choice was made erroneously.

5. Confirm this action by pressing the **ENTER** key or, if this is not the desired action, press the **EXIT** key.

After the **ENTER** key is pressed, the instrument automatically erases all stored data and returns to the last used program.

SECTION 6 PRINTING AND DATA TRANSFER

6.1 Data Transfer Adapter Basics

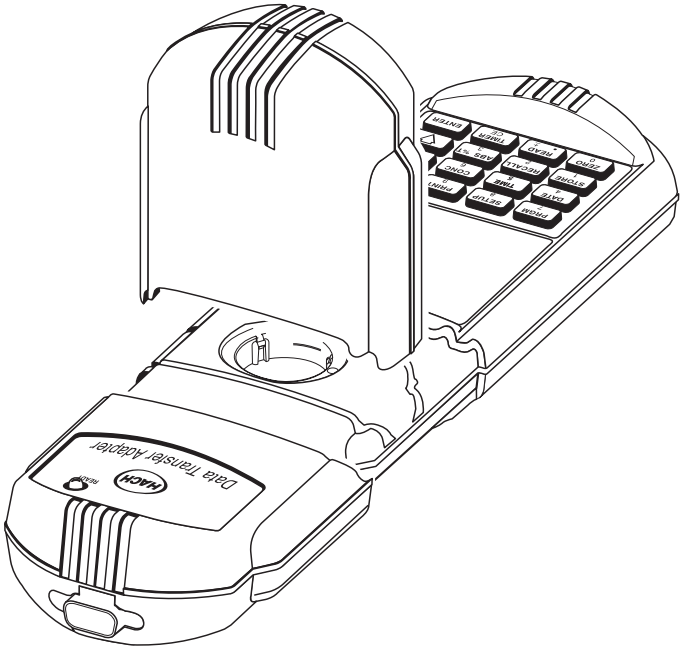
The optional Data Transfer Adapter (DTA) utilizes infra-red technology to receive data from the DR/800 Series Colorimeter and then transfer the signal into RS232 format. The DTA then sends the RS232 signal to a printer or personal computer.

The sleeve design makes the adapter compact, easy to use, and robust. Samples may be tested while the adapter is in place and the information may be immediately printed or downloaded to a computer. Data stored in the instrument memory may also be printed or downloaded at any time.

6.1.1 Attaching the Data Transfer Adapter

The Data Transfer Adapter is designed to fit on the instrument in the same way the instrument cap does. To install the DTA, simply remove the instrument cap, then slide the DTA onto the colorimeter body until it snaps into place. The DTA design allows the instrument cap to be used as a light shield. See *Figure 9 Installing the Data Transfer Adapter*.

Figure 9 Installing the Data Transfer Adapter



6.2 RS232 Connections

The RS232 receptacle on the Data Transfer Adapter connects with a 9-pin Sub-D connector (see *Figure 10 RS232 Connection*). A suitable RS232 cable is listed under Optional Accessories in *REPLACEMENT PARTS* on page *69*.

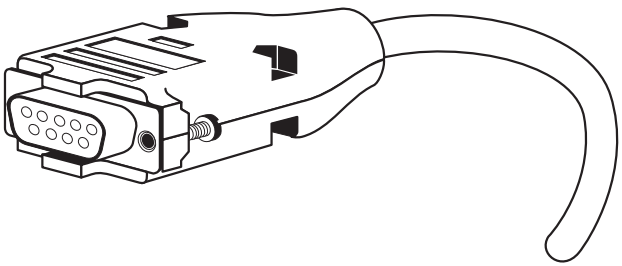
The RS232 interface output is an eight-bit data word plus one stop bit and no parity with a baud rate of 1200. It can communicate with either a serial printer or a serial communication port on a computer.

Press the **PRINT** key to send data to the printer or computer. See Section *6.3 Sending Data to a Printer or Computer* for instructions.

With the use of a serial-to-parallel converter, the data string transmitted from the colorimeter prints on any compatible parallel printer of the type normally used with IBM compatible applications.

All RS232 connections are made using the serial I/O port on the DTA. This port uses an industry standard 9-pin connector. See *Figure 10 RS232 Connection*.

Figure 10 RS232 Connection



Note: *For optimum performance and ESD protection, use a five-conductor shielded cable. Use a metal shell for the printer or CRT terminal connector, and connect the shield of the cable to the metal shell and to the sleeve (signal ground) of the RS232 plug.*

6.2.1 Setup and Use of the Printer

Follow the manufacturer's instructions when configuring the printer for compatibility with the colorimeter.

Pressing **PRINT** manually starts the printing, and pressing the **EXIT** key stops the printing (refer to Section *6.3 Sending Data to a Printer or Computer).*

Connect the DTA to a printer using the printer interface cable listed in *REPLACEMENT PARTS* on page *69*. The cable provides a direct link between the instrument and the 9-pin connector used for the serial port on most serial printers.

Table 4 and *Table 5* show the proper pin connections for 9-pin computer cables and for 25-pin printer cables. Use of cables that do not match the pin information given may cause undesirable operation.

Table 4 Standard 9-pin to 9-pin Computer Cable

DR/800 Series 9-pin Connector Socket		Computer 9-pin D Connector, plug	
Pin	**Signal Name**	**Pin**	**Signal Name**
2	RXD	3	TXD
3	TXD	2	RXD
4	DTR	no connection	—
5	GND	5	GND
6	DSR	no connection	—
7	RTS	8	CTS
8	CTS	7	RTS

Table 5 Standard 9-pin to 25-pin Printer Cable

DR/800 Series 9-pin Connector Socket		Computer 9-pin D Connector, plug	
Pin	**Signal Name**	**Pin**	**Signal Name**
2	RXD	no connection	—
3	TXD	3	RXD
4	DTR	no connection	—
5	GND	7	GND

Table 5 Standard 9-pin to 25-pin Printer Cable

DR/800 Series 9-pin Connector Socket		Computer 9-pin D Connector, plug	
Pin	**Signal Name**	**Pin**	**Signal Name**
6	DSR	20	DTR
7	RTS	no connection	—
8	CTS	20	DTR

To print, the communication parameters (baud rate, data bits and parity) of the instrument and the printer must match.

6.2.2 Connecting to a Personal Computer

Connect the colorimeter to a personal computer (PC) with the computer interface cable (Cat. No. 48129-00) listed under *REPLACEMENT PARTS* on page *69*. The cable provides a direct link between the colorimeter and the 9-pin D connector used for the serial port on most personal computers. If your computer has a 25-pin D connector, use a 9-pin to 25-pin adapter (available at most computer supply stores).

Use a communications software, such as HachLink™ Software (Cat. No. 49665-00) to collect data from the instrument. HachLink is a Windows-based application that allows a personal computer to capture data from several Hach instruments, including the DR/800 Series Colorimeters.

The captured data can be stored in a text file as a spread-sheet compatible format or as free-format text. Data captured in the spreadsheet format is easily transferred into most spreadsheet programs (i.e., Excel®, Microsoft Works®, Lotus 123®) for graphing and reporting.

To install and run HachLink™ Software, the computer and software must meet the following minimum requirements:

- IBM PC/AT or compatible with a 386SX processor (16 MHz or better)
- 4 megabytes of RAM
- Hard disk drive with 2 megabytes or more of free space

- 3-1/2 inch, 1.44 megabyte floppy disk drive
- VGA graphics with 640 x 480 or higher resolution, 16 or more colors
- Mouse or other pointing device
- A 9-pin serial port (or 25-pin serial port with 9-pin adapter)
- Windows 3.1 or later
- DOS 3.3 or later

To transfer data, the communication parameters (baud rate, data bits and parity) of the instrument and the computer must match. Once the communication link is established, press **PRINT** to send data to the computer.

6.3 Sending Data to a Printer or Computer

A permanent record of test results may be obtained using the DTA RS232 serial output to drive a printer or by transferring the data to a computer for storage. Data displayed on the instrument screen may be sent to an accessory printer or computer by attaching the DTA to the instrument and pressing the **PRINT** key. The data can be data recalled from memory or the current sample reading. Only the information shown on the display will be printed.

6.3.1 Sending Currently Displayed Data

To transfer currently displayed data:

1. Remove the instrument cap and slide the DTA onto the instrument until snug.
2. Make sure the DTA is properly connected to the computer or printer. See *Section 6.2 RS232 Connections.*
3. Press the **PRINT** key. Four dashes and **PRINT** are displayed while the data is being transmitted to the DTA.

6.3.2 Sending Recalled Data

To transfer recalled data:

1. Remove the instrument cap and slide the DTA onto the instrument until snug.
2. Make sure the DTA is properly connected to the computer or printer. (See *Section 6.2 RS232 Connections*.)
3. Recall the data to be transferred (see *Section 5.1 Recalling Data* on page *43*).
4. When sample data is displayed, press the **PRINT** key. Four horizontal lines and **PRINT** are displayed while the data is transmitted to the DTA.

6.3.3 Sending All Stored Data

All data stored in memory may be sent to a printer or computer via the **SETUP** menu option as follows:

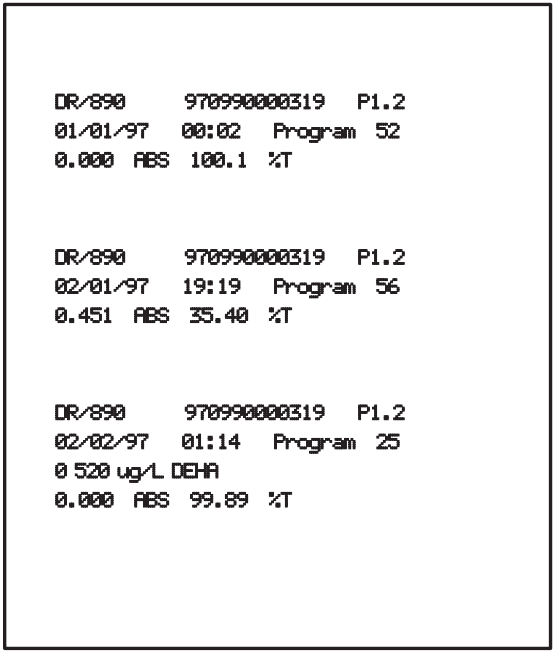
1. Remove the instrument cap and slide the DTA onto the instrument until snug.
2. Make sure the DTA is properly connected to the computer or printer. (See *Section 6.2 RS232 Connections*.)
3. Press the **SETUP** key.
4. Scroll, using the down **ARROW** key, to the **PRINT** option.
5. Press the **ENTER** key when the word **ALL** is displayed above **PRINT**. All stored data is now sent to the DTA. After the data has been successfully sent, the instrument defaults to the last used program.

Transferred data will include the following information:

- instrument model number
- instrument serial number
- instrument software version
- date

- time
- program number
- sample number
- concentration
- units
- chemical form
- Overrange errors (limit)
- absorbance
- %T

Figure 11 Printed Data Format



SECTION 7 MAINTENANCE

7.1 Cleaning the Colorimeter

Use a damp cloth to wipe the outside of the colorimeter enclosure. Wipe up spills promptly. Use cotton swabs to clean and dry the sample compartment if any spillage occurs.

Keep the colorimeter and sample cells clean at all times. Use a lens tissue or a soft, lint-free cloth (that will not leave an oil film) to wipe out the sample cell.

7.1.1 Cleaning the Data Transfer Adapter

Little cleaning is required of this adapter. Clean the outside and inside with a barely damp cloth. Wipe up spills promptly.

7.1.2 Sample Cells

Clean sample cells with detergent, rinse several times with tap water, and then rinse thoroughly with deionized water. Some cells may require acid washing or other special cleaning procedures. Refer to the Procedures Manual for additional information. Rinse sample cells used with organic solvents (chloroform, benzene, toluene, etc.) with acetone before the detergent wash, and again as a final rinse before drying.

7.2 Replacement Instructions

To prevent static electricity damage to the instrument, always turn the instrument off before removing the batteries.

7.2.1 Battery Replacement

When the **LOW BATTERY** icon appears in the display the batteries must be replaced or recharged as soon as possible to ensure proper instrument performance. Turn the instrument off before removing the battery compartment door.

The correct date and time may need to be reentered after battery replacement. See *Section 2.3 Setting the Date and Time*.

See *Section 2.1 Battery Installation* for complete installation instructions.

SECTION 8 TROUBLESHOOTING

8.1 Introduction

Correcting problem conditions with the DR/800 Series Colorimeters in the field is limited to responding to the error messages presented in the display. Other problems must be handled by a Hach technician at a service center. Refer to *REPAIR SERVICE*. **Do not** attempt to service anything other than the battery; there are no other field-serviceable parts. Opening the instrument case will void the warranty.

8.1.1 Error Codes

This feature identifies the problem area or areas when an error indication occurs during instrument operation. When an error occurs, **ERROR** displays on the screen, followed by a number which refers to a diagnostic error code. Refer to *Table 6* below to determine the error cause and possible corrective actions. Turn the instrument off, then on, to restore the instrument to operation.

Table 6 Error Codes

ERROR Code Number	Error Code Type	Corrective Action
1	Unconfigured instrument	Contact Hach Instrument Service Department
2	Could not read program data	Contact Hach Instrument Service Department
3	Could not write program data	Contact Hach Instrument Service Department
4	Measurement battery error	Replace instrument batteries
5	Measurement A/D error	Contact Hach Instrument Service Department
6	Measurement offset error	Check to be sure instrument light shield (cap) is correctly installed
7	Measurement low light error	Check for light path blockage Zero is out of instrument range; dilute to within range Contact Hach Instrument Service Department
8	Measurement over-range error	Make sure instrument cap is properly installed Contact Hach Instrument Service Department

8.1.2 Beeper/Error Icon

When a key is pressed that calls for the instrument to perform a function that is not available at that time, the beeper is activated once and the error icon appears on the display. Each time the unacceptable command is entered, one beep sounds.

If an unavailable program number is entered, the beeper also sounds. Program numbers must range from 101 to 105 (user programs) and 1 to 100 (Hach programs). Entering a number other than these results in the beeper sound and the error icon briefly illuminates. After this beep sounds, re-enter the proper number.

8.1.3 Concentration Out of Range

An out-of-range condition is indicated by the illuminated **LIMIT** icon. It means the sample concentration exceeds the range of the programmed calibration. Make sure the test procedure is followed correctly, dilute the sample (for over range samples) and rerun the test. Each Hach test has an upper concentration value that defines the program range. Measurements beyond that range are unreliable.

8.1.4 Low Battery

The instrument continuously monitors battery voltage. If the battery voltage falls to a level which indicates less than ten percent battery life remains, the instrument automatically warns the operator by displaying the **LOW BATTERY** icon. Replace the batteries as soon as possible for most reliable instrument performance.

APPENDIX A AVAILABLE PARAMETERS AND RANGES

Table 7 DR/820 Chemistries

PARAMETER	Primary Form	Alternate Forms	Test Range of Primary Form (mg/L or as noted)	Program number
Aluminum, Aluminon	Al	$Al_2 0_3$	0 - 0.800	1
Bromine	Br_2	—	0 - 4.50	5
Bromine, AV	Br_2	—	0 - 4.50	6
Chlorine, free, HR	Cl_2	—	0 - 5.00	8
Chlorine, total, HR	Cl_2	—	0 - 5.00	8
Chlorine, free	Cl_2	—	0 - 2.00	9
Chlorine, total	Cl_2	—	0 - 2.00	9
Chlorine, free, AV	Cl_2	—	0 - 2.00	11
Chlorine, total, AV	Cl_2	—	0 - 2.00	11
Chlorine, free, Test 'N Tube	Cl_2	—	0 - 5.00	10
Chlorine, total, Test 'N Tube	Cl_2	—	0 - 5.00	10
Chlorine Dioxide	ClO_2	—	0 - 5.00	112
Chlorine Dioxide, AV	ClO_2	—	0 - 5.00	113
COD, Manganese III	COD	—	20 - 1000	18
Cyanuric acid	CYACD	—	0 - 55	24
Hardness, calcium	$CaCO_3$	Ca	0 - 4.00	29
Hardness, magnesium	$CaCO_3$	Mg, $MgCO_3$	0 - 4.00	30
Iron, Ferrous	Fe	—	0 - 3.00	33
Iron, Ferrous, AV	Fe	—	0 - 3.00	33
Iron, total, FerroVer	Fe	—	0 - 3.00	33
Iron, total, FerroVer, AV	Fe	—	0 - 3.00	33
Manganese, HR	Mn	MnO_4, $KMnO_4$	0 - 20.0	41
Nitrate, HR, AV	NO_3-N	NO_3	0 - 30.0	50
Nitrate, HR	NO_3-N	NO_3	0 - 30.0	51
Nitrate, LR	NO_3-N	NO_3	0 - 0.50	55
Nitrite, LR	NO_2-N	NO_2, $NaNO_2$	0 - 0.350	60
Nitrite, LR, AV	NO_2-N	NO_2, $NaNO_2$	0 - 0.350	62
Nitrite, TNT	NO_2-N	NO_2, $NaNO_2$	0 - 0.500	63
Oxygen, dissolved, HR, AV	O_2	—	0 - 15.0	70
pH	pH	—	6.5 - 8.5 pH	75
Phosphorous, amino acid	PO_4	P, P_2O_5	0 - 30.0	85
Sulfate	SO_4	—	0 - 70	91
Sulfate, AV	SO_4	—	0 - 70	92
Turbidity	FAU	—	0 - 1000 FAU	95
Volatile Acids	HOAc	—	0 - 2800	96

Table 8 DR/850 Chemistries

PARAMETER	Primary Form	Alternate Forms	Test Range of Primary Form (mg/L or as noted)	Program number
Aluminum, Aluminon	Al	Al_20_3	0 - 0.800	1
Bromine	Br_2	—	0 - 4.50	5
Bromine, AV	Br_2	—	0 - 4.50	6
Chlorine, free, HR	Cl_2	—	0 - 5.00	8
Chlorine, total, HR	Cl_2	—	0 - 5.00	8
Chlorine, free	Cl_2	—	0 - 2.00	9
Chlorine, total	Cl_2	—	0 - 2.00	9
Chlorine, free, AV	Cl_2	—	0 - 2.00	11
Chlorine, total, AV	Cl_2	—	0 - 2.00	11
Chlorine, free, Test 'N Tube	Cl_2	—	0 - 5.00	10
Chlorine, total, Test 'N Tube	Cl_2	—	0 - 5.00	10
Chlorine Dioxide	ClO_2	—	0 - 5.00	112
Chlorine Dioxide, AV	ClO_2	—	0 - 5.00	113
COD, HR, HR+	COD	—	0 - 1500, 0-15,000	17
COD, Manganese III	COD	—	20 - 1000	18
Cyanide	CN	—	0 - 0.240	23
Cyanuric acid	CYACD	—	0 - 55	24
Detergents	LAS	—	0 - 0.30	26
Fluoride, SPADNS	F	—	0 - 2.00	27
Fluoride, SPADNS, AV	F	—	0 - 2.0	28
Hardness, calcium	$CaCO_3$	Ca	0 - 4.00	29
Hardness, magnesium	$CaCO_3$	Mg, $MgCO_3$	0 - 4.00	30
Iron, Ferrous	Fe	—	0 - 3.00	33
Iron, Ferrous, AV	Fe	—	0 - 3.00	33
Iron, total, FerroVer	Fe	—	0 - 3.00	33
Iron, total, FerroVer, AV	Fe	—	0 - 3.00	33
Iron, total, FerroMo	Fe	—	0 - 1.80	38
Iron, total, TPTZ	Fe	—	0 - 1.80	39
Iron, total, TPTZ, AV	Fe	—	0 - 1.80	39
Manganese, HR	Mn	MnO_4, $KMnO_4$	0 - 20.0	41
Molybdenum, ternary complex	Mo^6	MoO_4	0 - 3.00	47
Nitrogen, monochloramine and free ammonia, Salicylate	N	Cl_2, NH_3	0 - 0.50	49

Table 8 DR/850 Chemistries (Continued)

PARAMETER	Primary Form	Alternate Forms	Test Range of Primary Form (mg/L or as noted)	Program number
Nitrogen, monochloramine and free ammonia, Salicylate, AV	N	Cl_2, NH_3	0 - 0.50	49
Nitrate, HR, AV	NO_3-N	NO_3	0 - 30.0	50
Nitrate, HR	NO_3-N	NO_3	0 - 30.0	51
Nitrate, LR	NO_3-N	NO_3	0 - 0.50	55
Nitrite, LR	NO_2-N	NO_2, $NaNO_2$	0 - 0.350	60
Nitrite, LR, AV	NO_2-N	NO_2, $NaNO_2$	0 - 0.350	62
Nitrite, TNT	NO_2-N	NO_2, $NaNO_2$	0 - 0.500	63
Nitrogen, Ammonia, Salicylate	NH_3-N	NH_3 , NH_4	0 - 0.50	64
Nitrogen, Ammonia, LR, TNT	NH_3-N	NH_3	0 - 2.50	66
Nitrogen, Ammonia, HR, TNT	NH_3-N	NH_3	0 - 50	67
Nitrogen, Total Inorganic, TNT	N	NH_3	0 - 25.0	68
Oxygen, dissolved, HR, AV	O_2	—	0 - 15.0	70
Oxygen, dissolved, LR, AV	O_2	—	0 - 1000 µg/L	71
Ozone, LR, AV	O_3	—	0 - 0. 25	72
Ozone, MR, AV	O_3	—	0 - 1.50	73
Ozone, HR, AV	O_3	—	0 - 0.75	74
pH	pH	—	6.5 - 8.5 pH	75
Phosphonates	PO_4	—	0-125	80
Phosphorous, PhosVer 3	PO_4	P, P_2O_5	0 - 2.50	79
Phosphorous, PhosVer 3, AV	PO_4	P, P_2O_5	0 - 2.50	79
Phosphorous, total, PhosVer 3	PO_4	P, P_2O_5	0 - 2.5	79
Phosphorous, acid hydrolyzable, PhosVer 3	PO_4	P, P_2O_5	0 - 2.5	79
Phosphorous, PhosVer 3, TNT	PO_4	P, P_2O_5	0 - 5.0	82
Phosphorous, total, PhosVer 3, TNT	PO_4	P, P_2O_5	0 - 3.50	82
Phosphorous, acid hydrolyzable, PhosVer 3, TNT	PO_4	P, P_2O_5	0 - 5.00	82
Phosphorous, amino acid	PO4	P, P_2O_5	0 - 30	85
Silica, LR	SiO2	—	0 - 1.60	90
Sulfate	SO_4	—	0 - 70	91
Sulfate, AV	SO_4	—	0 - 70	92
Sulfide	S	—	0 - 0.70	93

Table 8 DR/850 Chemistries (Continued)

PARAMETER	Primary Form	Alternate Forms	Test Range of Primary Form (mg/L or as noted)	Program number
Suspended Solids	SuSld	—	0 - 750	94
Tannin and Lignin	tanic	—	0 - 9.0	98
Toxicity	Toxic	—	0 - 100% Inhibition	61
Turbidity	FAU	—	0 - 1000 FAU	95
Volatile Acids	HOAc	—	0 - 2800	96
Zinc	Zn	—	0 - 3.00	97

Table 9 DR/890 Chemistries

PARAMETER	Primary Form	Alternate Forms	Test Range of Primary Form (mg/L or as noted)	Program number
Aluminum, Aluminon	Al	Al_20_3	0 - 0.800	1
Boron	B	H_3BO_3	0 - 1.60	4
Bromine	Br_2	—	0 - 4.50	5
Bromine, AV	Br_2	—	0 - 4.50	6
Chlorine Dioxide, MR	ClO_2	—	0 - 50	7
Chlorine, free, HR	Cl_2	—	0 - 5.00	8
Chlorine, total, HR	Cl_2	—	0 - 5.00	8
Chlorine, free	Cl_2	—	0 - 2.00	9
Chlorine, total	Cl_2	—	0 - 2.00	9
Chlorine, free, AV	Cl_2	—	0 - 2.00	11
Chlorine, total, AV	Cl_2	—	0 - 2.00	11
Chlorine, free, Test ‘N Tube	Cl_2	—	0 - 5.00	10
Chlorine, total, Test ‘N Tube	Cl_2	—	0 - 5.00	10
Chlorine Dioxide	ClO_2	—	0 - 5.00	112
Chlorine Dioxide, AV	ClO_2	—	0 - 5.00	113
Chromium, Hexavalent	Cr^6	CrO_4, Cr_2O_7	0 - 0.60	13
Chromium, Hexavalent, AV	Cr^6	CrO_4, Cr_2O_7	0 - 0.60	14
Chromium, total	Cr	—	0 - 0.60	15
COD, LR	COD	—	0 - 150	16
COD, HR, HR+	COD	—	0 - 1500, 0 - 15000	17
COD, Manganese III	COD	—	20 - 1000	18
Color	Pt Co	—	0 - 500 APHA color	19
Copper, Bichinchoninate	Cu	—	0 - 5.00	20

Table 9 DR/890 Chemistries (Continued)

PARAMETER	Primary Form	Alternate Forms	Test Range of Primary Form (mg/L or as noted)	Program number
Copper, Bichinchoninate, AV	Cu	—	0 - 5.00	21
Copper, porphyrin	Cu	—	0 - 210.0 µg/L	22
Cyanide	CN	—	0 - 0.240	23
Cyanuric acid	CYACD	—	0 - 55	24
DEHA	DEHA	—	0 - 500 µg/L	25
Detergents	LAS	—	0 - 0.30	26
Fluoride, SPADNS	F	—	0 - 2.00	27
Fluoride, SPADNS, AV	F	—	0 - 2.00	28
Hardness, calcium	$CaCO_3$	Ca	0 - 4.00	29
Hardness, magnesium	$CaCO_3$	Mg, $MgCO_3$	0 - 4.00	30
Hydrazine	N_2H_4	—	0 - 500 µg/L	31
Hydrazine, AV	N_SH_4	—	0 - 500 µg/L	32
Immunoassay, PCB	—	—	threshold	42
Immunoassay, TPH	—	—	threshold	42
Immunoassay, TPH in water	—	—	threshold	42
Iron, Ferrous	Fe	—	0 - 3.00	33
Iron, Ferrous, AV	Fe	—	0 - 3.00	33
Iron, total, FerroVer	Fe	—	0 - 3.00	33
Iron, total, FerroVer, AV	Fe	—	0 - 3.00	33
Iron, total, Ferrozine	Fe	—	0 - 1.300	37
Iron, total, FerroMo	Fe	—	0 - 1.80	38
Iron, total, TPTZ	Fe	—	0 - 1.80	39
Iron, total, TPTZ, AV	Fe	—	0 - 1.80	39
Manganese, HR	Mn	MnO_4, $KMnO_4$	0 - 20.0	41
Manganese, LR	Mn	MnO_4, $KMnO_4$	0 - 0.700	43
Molybdenum, Molybdate, HR	Mo^6	MoO_4	0 - 40.0	44
Molybdenum, Molybdate, HR, AV	Mo^6	MoO_4	0 - 40.0	44
Molybdenum, ternary complex	Mo^6	MoO_4	0 - 3.00	47
Nickel, PAN	Ni	—	0 - 1.000	48
Nitrogen, monochloramine and free ammonia, Salicylate	N	Cl_2, NH_3	0 - 0.50	49
Nitrogen, monochloramine and free ammonia, Salicylate, AV	N	Cl_2, NH_3	0 - 0.50	49
Nitrate, HR, AV	NO_3-N	NO_3	0 - 30.0	50

Table 9 DR/890 Chemistries (Continued)

PARAMETER	Primary Form	Alternate Forms	Test Range of Primary Form (mg/L or as noted)	Program number
Nitrate, HR	NO_3-N	NO_3	0 - 30.0	51
Nitrate, Cd reduction, MR, AV	NO_3-N	NO_3	0 - 5.0	53
Nitrate, Cd reduction, MR	NO_3-N	NO_3	0 - 5.0	54
Nitrate, LR	NO_3-N	NO_3	0 - 0.50	55
Nitrate, TNT, chromotropic acid finish	NO_3-N	NO_3	0 - 30.0	57
Nitrogen, TN, TNT, chromotropic acid	N	NO_3, NH_3	0 - 25	58
Nitrite, HR	NO_2	NO_2-N, $NaNO_2$	0 - 150	59
Nitrite, LR	NO_2-N	NO_2, $NaNO_2$	0 - 0.350	60
Nitrite, LR, AV	NO_2-N	NO_2, $NaNO_2$	0 - 0.350	62
Nitrite, TNT	NO_2-N	NO_2, $NaNO_2$	0 - 0.500	63
Nitrogen, Ammonia, Salicylate	NH_3-N	NH_3 , NH_4	0 - 0.50	64
Nitrogen, TKN with Nessler finish	TKN	—	0 - 150	65
Nitrogen, Ammonia, LR, TNT	NH_3-N	NH_3	0 - 2.50	66
Nitrogen, Ammonia, HR, TNT	NH_3-N	NH_3	0 - 50	67
Nitrogen, Total Inorganic TNT	N	NH_3	0 - 25.0	68
Nitrogen, Total, HR, TNT	N	NH_3	10 - 150	69
Oxygen, dissolved, HR, AV	O_2	—	0 - 15.0	70
Oxygen, dissolved, LR, AV	O_2	—	0 - 1000 µg/L	71
Ozone, LR, AV	O_3	—	0 - 0.25	72
Ozone, MR, AV	O_3	—	0 - 0.75	73
Ozone, HR, AV	O_3	—	0 - 1.50	74
pH	pH	—	6.5 - 8.5 pH	75
Phosphonates	PO_4	—	0 - 125	80
Phosphorous, Molybdovanadate	PO_4	P, P_2O_5	0 - 45.0	77
Phosphorous, Molybdovanadate, AV	PO_4	P, P_2O_5	0 - 45.0	78
Phosphorous, PhosVer 3	PO_4	P, P_2O_5	0 - 2.50	79
Phosphorous, PhosVer 3, AV	PO_4	P, P_2O_5	0 - 2.50	79
Phosphorous, total, PhosVer 3	PO_4	P, P_2O_5	0 - 2.5	79
Phosphorous, acid hydrolyzable, PhosVer 3	PO_4	P, P_2O_5	0 - 2.5	79
Phosphorous, PhosVer 3, TNT	PO_4	P, P_2O_5	0 - 5.0	82

Table 9 DR/890 Chemistries (Continued)

PARAMETER	Primary Form	Alternate Forms	Test Range of Primary Form (mg/L or as noted)	Program number
Phosphorous, total, PhosVer 3, TNT	PO_4	P, P_2O_5	0 - 3.50	82
Phosphorous, acid hydrolyzable, PhosVer 3, TNT	PO_4	P, P_2O_5	0 - 5.00	82
Phosphorous, amino acid	PO_4	P, P_2O_5	0 - 30.0	85
Phosphorus, Reactive, HR, TNT	PO_4^{3-}	P, P_2O_5	0 - 100.0	86
Phosphorus, Total, HR, TNT	PO_4^{3-}	P, P_2O_5	0 - 100.0	87
Silica, UHR	SiO_2	—	0 - 200	88
Silica, HR	SiO_2	—	0 - 75.0	89
Silica, LR	SiO_2	—	0 - 1.60	90
Sulfate	SO_4	—	0 - 70	91
Sulfate, AV	SO_4	—	0 - 70	92
Sulfide	S	—	0 - 0.70	93
Suspended Solids	SuSld	—	0 - 750	94
Triazole, Benzotriazole	BENZO	TOLY	0 - 16.0	3
Triazole, Tolytriazole	TOLY	BENZO	0 - 16.0	3
Tannin and Lignin	Tanic	—	0 - 9.0	98
Toxicity	Toxic	—	0 - 100% Inhibition	61
Turbidity	FAU	—	0 - 1000 FAU	95
Volatile Acids	HOAc	—	0 - 2800	96
Zinc	Zn	—	0 - 3.00	97

GENERAL INFORMATION

At Hach Company, customer service is an important part of every product we make.

With that in mind, we have compiled the following information for your convenience.

REQUIRED APPARATUS

Description	Unit	Cat. No.
Adapter, Assembly, COD	each	48464-00
Batteries, Alkaline AA	pkg/4	19380-04
Battery Cover Assembly	each	48455-00
Battery Holder for 4 Alkaline AA cells	each	48434-00
Manual Set, DR/890, includes Instrument & Procedure Manual & Binder	each	48470-77
Manual Set, DR/850, includes Instrument & Procedure Manual & Binder	each	48450-77
Manual Set, DR/820, includes Instrument & Procedure Manual & Binder	each	48440-77
Sample Cell, 25 x 95 mm 10-20-25 mL	pkg/6	24019-06

OPTIONAL ACCESSORIES

Adapter, Immunoassay	each	48467-00
Adapter, Data Transfer, RS232, includes 48129-00 cable	each	48490-00
Batteries, Rechargeable, NiCad, for PN60 Printer	each	26688-00
Batteries, Rechargeable, Alkaline AA, for DR/800 Series Colorimeter	pkg/4	49427-00
Battery Charger, Alkaline AA 115 VAC UL Approved	each	49428-00
Cap, Sample Cell, for 25 x 95 mL cell	pkg/12	24018-12
Carrying Case, DR/800 Series Colorimeter, hard-sided	each	49425-00
Carrying Case, DR/800 Series Colorimeter, soft-sided w/shoulder strap	each	27220-00
Carrying Case, Portable Laboratory	each	49430-00
Computer Interface Cable, 6 ft., for use with the DTA	each	48129-00
DR/Check™ ABS Standards	set	27639-00
Foot, Rubber, DR/800 Series Colorimeter	each	49424-00
HachLink Software	each	49665-00
Instrument Cap	each	49431-00
Power Cord for PN60 Printer, European Plug	each	46836-00
Printer, 115/230V, Citizen PN60*	each	26687-00
Printer Cable Assembly	each	26689-00
Printer Ink Cartridge, for PN60 Printer, Black	pkg/2	26690-00

* Requires Data Transfer Adapter

HOW TO ORDER

How To Order Within The United States

By phone (in U.S.A.):
6:30 a.m. to 5 p.m. MST
Monday through Friday
800-227-Hach (800-227-4224)
970-669-3050 (Hach Loveland)
By Telex: 160840 (Hach Loveland)

By mail:
Hach Company
P.O. Box 389
Loveland, Colorado 80539-0389
U.S.A.
By FAX: 970-669-2932
(Hach Loveland)

How To Order Outside The United States

Hach maintains a worldwide network of dealers and distributors. Contact your local distributor for help.

In Europe, Mediterranean Africa, and the Middle East:

Hach Europe, S.A./N.V.
Chaussée de Namur, 1
B-5150 Floriffoux (Namur), Belgium
Telephone: (32)(81)44.71.71
FAX: (32)(81)44.13.00

In other areas, obtain assistance from a local Hach distributor or:

Hach Company World Headquarters
P.O. Box 389
Loveland, Colorado 80539-0389
U.S.A.
Telephone: (970) 669-3050
FAX: (970) 669-2932

Hach Sales & Service Canada Ltd.
1313 Border Street, Unit 34
Winnipeg, Manitoba R3H 0X4
Telephone: (204) 632-5598
FAX: (204) 694-5134

Information Required

- Hach Account number
- Billing Address
- Your Name and phone number
- Purchase order number
- Catalog number
- Brief description or model number
- Quantity

REPAIR SERVICE

Authorization must be obtained from Hach Company before sending any item for repair. Please contact the Hach Factory Service Center serving your location.

In the United States:

HACH COMPANY
100 Dayton Ave.
P.O. Box 907
Ames, Iowa 50010
800-227-4224 (U.S.A. only)
FAX: (515) 232-1276

In Latin America, the Caribbean, the Far East, the Indian subcontinent, Africa (excluding Mediterranean Africa) or the Pacific Basin:

HACH COMPANY, WORLD HEADQUARTERS
P.O. Box 389
Loveland, Colorado 80539-0389
U.S.A.
Telephone (970) 669-3050
Telex 160840
FAX (970) 669-2932

In Canada:

HACH SALES & SERVICE CANADA LTD.
1313 Border Street, Unit 34
Winnipeg, Manitoba
R3H 0X4
800-665-7635 (Canada only)
(204) 632-5598
FAX: (204) 694-5134

In Europe, the Middle East, or Mediterranean Africa:

HACH EUROPE, S.A./N.V.
Chaussée de Namur, 1
B-51150 Floriffoux (Namur), Belgium
Tel. 32-(0)81-44.71.71

WARRANTY

Hach warrants the DR/800 Series Colorimeters against defective materials or workmanship for one year from the date of shipment.

HACH WARRANTS TO THE ORIGINAL BUYER THAT HACH PRODUCTS WILL CONFORM TO ANY EXPRESS WRITTEN WARRANTY GIVEN BY HACH TO THE BUYER. EXCEPT AS EXPRESSLY SET FORTH IN THE PRECEDING SENTENCE, HACH MAKES NO WARRANTY OF ANY KIND WHATSOEVER WITH RESPECT TO ANY PRODUCTS.
HACH EXPRESSLY DISCLAIMS ANY WARRANTIES IMPLIED BY LAW, INCLUDING BUT NOT LIMITED TO ANY WARRANTY OF MERCHANT-ABILITY OR FITNESS FOR A PARTICULAR PURPOSE.

LIMITATION OF REMEDIES: Hach shall, at its option, replace or repair nonconforming products or refund all amounts paid by the buyer. **THIS IS THE EXCLUSIVE REMEDY FOR ANY BREACH OF WARRANTY.**

LIMITATION OF DAMAGES: IN NO EVENT SHALL HACH BE LIABLE FOR ANY INCIDENTAL OR CONSEQUENTIAL DAMAGES OF ANY KIND FOR BREACH OF ANY WARRANTY, NEGLIGENCE, ON THE BASIS OF STRICT LIABILITY, OR OTHERWISE.

Catalog descriptions, pictures and specifications, although accurate to the best of our knowledge, are not a guarantee or warranty.

For a complete description of Hach Company’s warranty policy, request a copy of our Terms and Conditions of Sale for U.S. Sales from our Customer Service Department.

CERTIFICATION

Hach Company certifies this instrument was tested thoroughly, inspected and found to meet its published specifications when it was shipped from the factory.

The DR/800 Series Colorimeter has been tested and is certified as indicated to the following instrumentation standards:

EN 60825-1: LEDs used in this product are Class 1

Immunity:

EN 50082-1 "1997"(Generic Immunity Standard) per **89/336/EEC EMC**: Supporting test records by Hach Company, certified compliance by Hach Company.

Required Standard/s include:
EN 61000-4-2 (IEC 1000-4-2) Electro-Static Discharge

EN 61000-4-3 (IEC 1000-4-3) Radiated RF Electro-Magnetic Fields

ENV 50204 Radiated Electro-Magnetic Field from Digital Telephones

Emissions:

Per **89/336/EEC EMC**: Supporting test records by Intellistor O.A.T.S., (NVLAP #0369) certified compliance by Hach Company.

Required European Standard/s include:
EN 55011 (CISPR 11) Emissions, Class B Limits

Additional Emissions Standard/s include:

CANADIAN INTERFERENCE-CAUSING EQUIPMENT REGULATION, IECS-003, Class A:
Supporting test records by Intellistor O.A.T.S., certified compliance by Hach Company.

This Class A digital apparatus meets all requirements of the Canadian Interference-Causing Equipment Regulations.

Cet appareil numérique de la classe A respecte toutes les exigences du Règlement sur le matériel brouilleur du Canada.

FCC PART 15, Class “A” Limits:

Supporting test records by Intellistor O.A.T.S., certified compliance by Hach Company.

This device complies with Part 15 of the FCC Rules. Operation is subject to the following two conditions:

(1) This device may not cause harmful interference, and (2) this device must accept any interference received, including interference that may cause undesired operation.

Changes or modifications to this unit not expressly approved by the party responsible for compliance could void the user’s authority to operate the equipment.

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. The following techniques of reducing the interference problems are applied easily.

1. Remove power from the Colorimeter by removing one of its batteries to verify that it is or is not the source of the interference.
2. Move the Colorimeter away from the device receiving the interference.
3. Reposition the receiving antenna for the device receiving the interference.
4. Try combinations of the above.

HACH COMPANY
WORLD HEADQUARTERS
P.O. Box 389
Loveland, Colorado 80539-0389
Telephone: (970) 669-3050
FAX: (970) 669-2932

HACH EUROPE
Chaussée de Namur, 1
B-5150 Floriffoux (Namur), Belgium
Telephone: (32)(81) 44.71.71
FAX: (32)(81) 44.13.00

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. - **Call toll-free 800-227-4224**
Outside the U.S.A. - **Contact the HACH office or distributor serving you.**
On the Worldwide Web - **http://www.hach.com**; E-mail - **techhelp@hach.com**

DR/4000 PROCEDURE

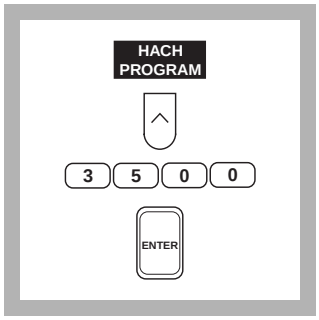
SULFIDE

✔ Method 8131 **Methylene Blue Method***

(0 to 800 µg/L)

Scope and Application: *For testing total sulfides, H_2S, HS^- and certain metal sulfides in groundwater, wastewater brines and seawater; USEPA accepted for reporting wastewater analysis***

* Adapted from *Standard Methods for the Examination of Water and Wastewater.*
** Procedure is equivalent to USEPA method 376.2 and Standard Method 4500-S^{2-} D for wastewater.



1. Press the soft key under ***HACH PROGRAM.***

Select the stored program number for sulfide (S^{2-}) by pressing **3500** with the numeric keys.

Press: **ENTER**

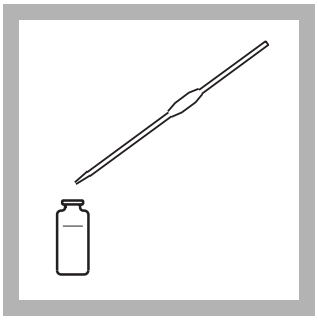
Note: *The Flow Cell and Sipper Modules can be used with this procedure.*

Note: *Samples must be analyzed immediately and cannot be preserved for later analysis. Avoid excessive agitation of samples.*

2. The display will show:

HACH PROGRAM: 3500 Sulfide

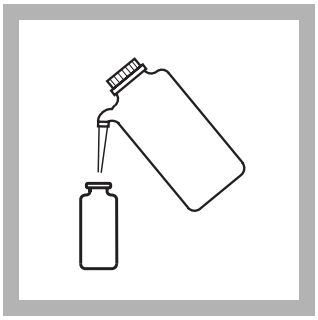
The wavelength (λ), **665 nm**, is automatically selected.



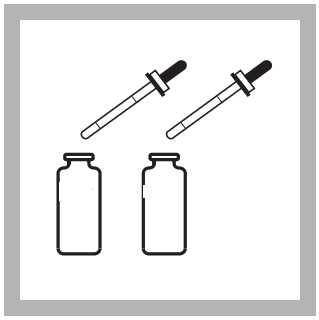
3. Measure 25 mL of sample into a sample cell. This will be the prepared sample.

Note: *For turbid samples, see Interferences (following these steps) for pretreatment instructions.*

Note: *Excessive agitation will cause loss of sulfide. Use a pipet to minimize sulfide loss.*

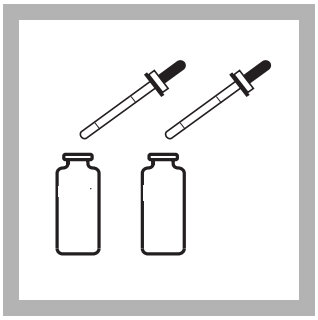


4. Measure 25 mL of deionized water into a second sample cell (the blank).



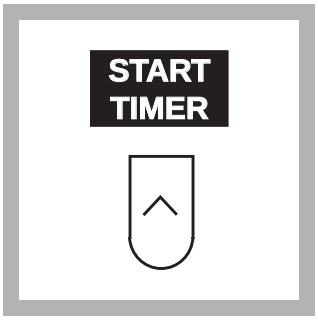
5. Add 1.0 mL of Sulfide 1 Reagent to each cell. Swirl to mix.

Note: *Use the calibrated 1-mL dropper.*



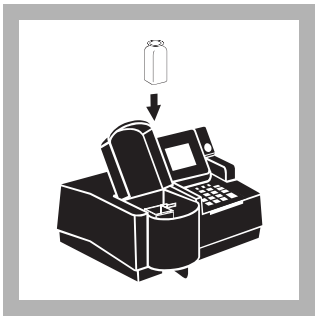
6. Add 1.0 mL of Sulfide 2 Reagent to each cell. Immediately swirl to mix.

Note: *A pink color will develop, then the solution will turn blue if sulfide is present.*

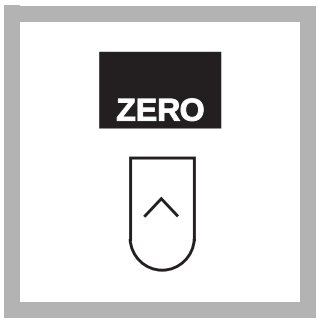


7. Press the soft key under ***START TIMER***.

A 5-minute reaction period will begin.



8. When the timer beeps, place the blank in the cell holder. Close the light shield.

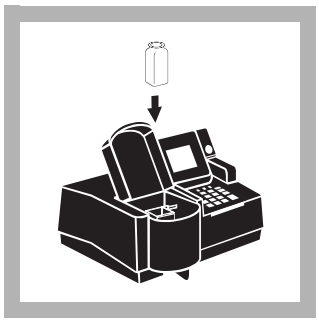


9. Press the soft key under ***ZERO***.

The display will show:

0 µg/L S^{2-}

Note: *For alternate concentration units, press the soft key under* ***OPTIONS****. Then press the soft key under* ***UNITS*** *to scroll through the available options. Press* **ENTER** *to return to the read screen.*



10. Place the prepared sample in the cell holder. Close the light shield. Results in µg/L sulfide (or chosen units) will be displayed.

Note: *Some sulfide loss may occur if dilution is necessary.*

Interferences

Table 1 Interfering Substances and Suggested Treatments

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 place of deionized water in step 4.

Sample Collection, Storage and Preservation

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation or prolonged exposure to air. Analyze samples immediately.

Method Performance

Precision
Standard: 400 µg/L S^{2-}

Program	95% Confidence Limits
3500	399–401 µg/L S^{2-}

For more information on determining precision data and method detection limits, refer to Section *1.5*.

Estimated Detection Limit

Program	EDL
3500	2 µg/L S^{2}

For more information on derivation and use of Hach's estimated detection limit, see Section *1.5.2*. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, appendix B, see Section *1.5.1*.

Sensitivity
Program Number: 3500

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	4.9 µg/L

See Section *1.5.3 Sensitivity Explained* for more information.

Determining 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 sulfate 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 proper dilution.

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. For additional information, refer to *Section 1*.

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. Please see *Section 1* for further information on proper disposal of these materials.

REQUIRED REAGENTS AND STANDARDS

	Cat. No.
Sulfide Reagent Set (100 tests)	22445-00

Includes: (2) 1816-32, (2) 1817-32

Description	Quantity Required per test	Unit	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 liters	272-56

REQUIRED EQUIPMENT AND SUPPLIES

Cylinder, graduated, 25-mL	1	each	508-40
or			
Pipet, volumetric, Class A, 25-mL	1	each	14515-40
DR/4000 1-Inch Cell Adapter	1	each	48190-00
Pipet Filler, safety bulb	1	each	14651-00

OPTIONAL REAGENTS AND STANDARDS

Bromine Water, 30-g/L	29 mL	2211-20
Phenol Solution, 30-g/L	29 mL	2112-20

OPTIONAL EQUIPMENT AND SUPPLIES

DR/4000 Carousel Module Kit	each	48070-02
DR/4000 Flow Cell Module Kit, 1-inch	each	48070-04
DR/4000 Flow Cell Module Kit, 1-cm	each	48070-05
DR/4000 Sipper Module Kit, 1-inch	each	48090-03
Dropper, for 1-oz. bottle	each	2258-00
Flask, Erlenmeyer, 50-mL	each	505-41

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

✔Method 8221

Buret Titration Method* **

Buret Titration (0 to 5,000 mg/L as $CaCO_3$)

Scope and Application: For water, wastewater, and seawater

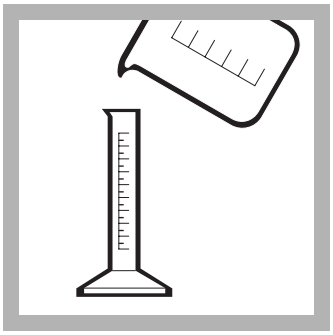
* Adapted from *Standard Method for the Examination of Water and Wastewater, 2320 B.*
** USEPA Accepted

Tips and Techniques

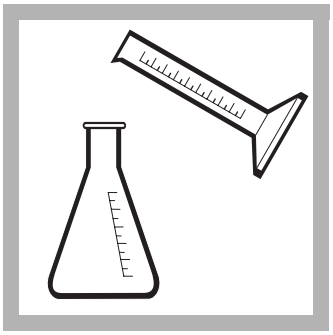
- A pH meter (Cat. No. 51700-10) is required for NPDES reporting and is recommended for best results.
- You can substitute six drops of Phenolphthalein Indicator Solution (Cat. No. 162-32) for the Phenolphthalein Indicator Powder Pillow.
- You can substitute six drops of Bromcresol Green-Methyl Red Indicator Solution (Cat. No. 23292-32) for the Bromcresol Green-Methyl Red Powder Pillow.
- mg/L as $CaCO_3$ ÷ 17.12 = grains per gallon

Buret Titration Method 8221

1. Select a sample size that corresponds to the expected alkalinity concentration in mg/L as calcium carbonate ($CaCO_3$) from *Table 1*.

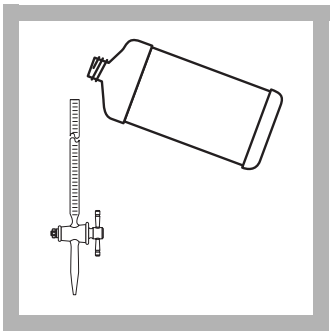


2. Use a graduated cylinder or pipet to measure the sample volume from *Table 1*.

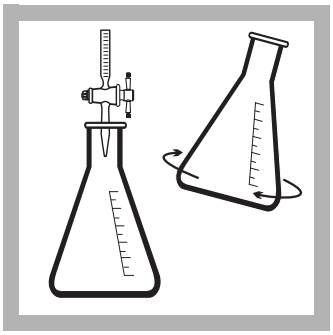


3. Transfer the sample into a 250-mL Erlenmeyer flask. If necessary, dilute to 50 mL with deionized water.

4. Add the contents of one Phenolphthalein Indicator Powder Pillow. Swirl to mix. Skip this step if you are using a pH meter.



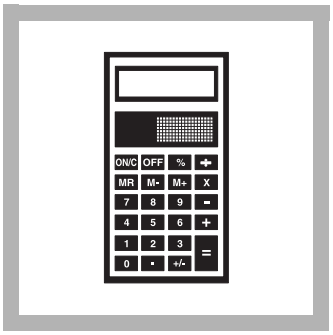
5. Fill a 25-mL buret to the zero mark with 0.020 N Sulfuric Acid Standard Solution.



6. Titrate the sample while swirling the flask until the solution changes from pink to colorless.

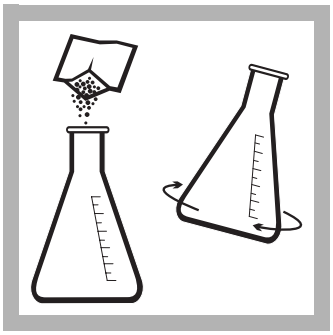
When using a pH meter, the end point is 8.3.

If the solution is colorless before titrating with sulfuric acid, the phenolphthalein alkalinity is zero.



7. Calculate:

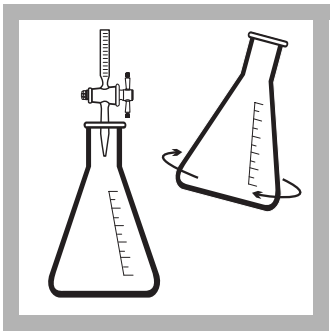
mL Titrant x Multiplier Used = mg/L Phenolphthalein Alkalinity as $CaCO_3$



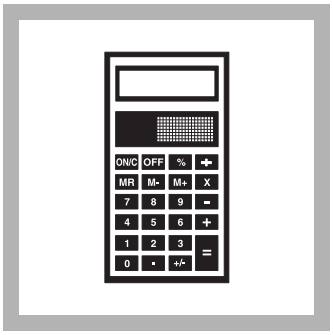
8. Add the contents of one Bromcresol Green-Methyl Red Indicator Powder Pillow to the titrated sample. Swirl to mix.

Do not add indicator if a pH meter is used.

Note: *Specific sample composition may require titration to a specific pH (see Table 3).*



9. Continue the titration until a light pink end point is reached.



10. Calculate:

mL Titrant x Multiplier Used = mg/L Total Alkalinity as $CaCO_3$

Table 1

Range (mg/L as $CaCO_3$)	Sample Volume (mL)	Titration Cartridge (N)	Catalog Number	Multiplier
0–500	50	0.020	203-53	20
400–1000	25	0.020	203-53	40
1000–2500	10	0.020	203-53	100
2000–5000	5	0.020	203-53	200

The end points in *Table 2* are recommended for determining total alkalinity in water samples of various compositions and alkalinity concentrations.

Table 2 Alkalinity Endpoints

Sample Composition	End Point pH	
	Total Alkalinity	Phenolphthalein Alkalinity
Alkalinity about 30 mg/L	pH 4.9	pH 8.3
Alkalinity about 150 mg/L	pH 4.6	pH 8.3
Alkalinity about 500 mg/L	pH 4.3	pH 8.3
Silicates or phosphates present	pH 4.5	pH 8.3
Industrial wastes or complex system	pH 4.5	pH 8.3
Routine or Automated Analyses	pH 4.5	pH 8.3

Total alkalinity primarily includes hydroxide, carbonate, and bicarbonate alkalinities. The concentration of these types in a sample may be determined when the phenolphthalein and total alkalinities are known. See *Table 3*.

Table 3 Alkalinity Relationship

Row	Result of Titration	Hydroxide Alkalinity Equals:	Carbonate Alkalinity Equals:	Bicarbonate Alkalinity Equals:
1	Phenolphthalein Alkalinity equal to 0	0	0	Total Alkalinity
2	Phenolphthalein Alkalinity equal to Total Alkalinity	Total Alkalinity	0	0
3	Phenolphthalein Alkalinity less than one-half of Total Alkalinity	0	Phenolphthalein Alkalinity times 2	Total Alkalinity minus two times Phenolphthalein Alkalinity
4	Phenolphthalein Alkalinity equal to one-half of Total Alkalinity	0	Total Alkalinity	0
5	Phenolphthalein Alkalinity greater than one-half of Total Alkalinity	2 times Phenolphthalein Alkalinity minus Total Alkalinity	2 times the difference between Total and Phenolphthalein Alkalinity	0

To use *Table 3*, follow these steps:

a. Does the phenolphthalein alkalinity equal zero? If yes, use Row 1.

b. Does the phenolphthalein alkalinity equal total alkalinity? If yes, use Row 2.

c. Divide the total alkalinity by 2 to calculate one-half the total alkalinity.

d. Select Row 3, 4 or 5 based on comparing the result of step c (one-half total alkalinity) with the phenolphthalein alkalinity.

e. Perform the required calculations if any.

f. Check your results. The sum of the three alkalinity types will equal the total alkalinity.

Example:

A sample has 170 mg/L as $CaCO_3$ phenolphthalein alkalinity and 250 mg/L as $CaCO_3$ total alkalinity. What is the concentration of hydroxide, carbonate, and bicarbonate alkalinities?

a. The phenolphthalein alkalinity does not equal zero but 170 mg/L.

b. The phenolphthalein alkalinity does not equal total alkalinity (170 mg/L vs. 250 mg/L).

c. One-half of the total alkalinity equals 125 mg/L.

d. Because the phenolphthalein alkalinity of 170 mg/L is greater than one-half the total alkalinity of 125 mg/L, select Row 5.

The hydroxide alkalinity is equal to:

2 x 170 = 340

340 - 250 = 90 mg/L hydroxide alkalinity

The carbonate alkalinity is equal to:

250 - 170 = 80

80 x 2 = 160 mg/L carbonate alkalinity

The bicarbonate alkalinity is equal to zero mg/L.

Check:

90 mg/L hydroxide alkalinity + 160 mg/L carbonate alkalinity + 0 mg/L bicarbonate alkalinity = 250 mg/L

The answer is correct.

The sum of each type equals the total alkalinity (250 mg/L).

Sampling and Storage

Collect samples in plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation and prolonged exposure to air. Samples should be analyzed as soon as possible after collection but can be stored at least 24 hours by cooling to 4 °C (39 °F) or below. Warm to room temperature before analyzing.

Accuracy Check

End Point Confirmation

- To accurately determine the phenolphthalein alkalinity end point, mix the contents of one Phenolphthalein Indicator Powder Pillow and the contents of one pH 8.3 Buffer Powder Pillow with 50 mL of deionized water in a 250-mL Erlenmeyer flask. The resulting color is the end point. The buffer solution without the indicator can be used to standardize a pH meter.
- To accurately determine the total alkalinity end point, mix the contents of one pH 4.5 Buffer Powder Pillow and the contents of one Bromcresol Green-Methyl Red Indicator Powder Pillow with 50 mL of deionized water in a 250-mL Erlenmeyer flask. Titrate to a light pink color change.

Standard Additions Method (Sample Spike)

Perform the standard additions method check as follows:

1. Snap the neck off an Alkalinity Voluette® Ampule Standard Solution, 0.500 N.
2. Use the TenSette Pipet (Cat. No. 19700-01) to add 0.1 mL of standard to the sample titrated in *step 6* or *step 9*. Resume titration back to the same end point. Record the volume of titrant needed.
3. Repeat, using two more additions of 0.1 mL. Titrate to the end point after each addition.
4. The mL of titrant required should increase by 2.5 mL for each 0.1 mL increment of standard added.

Interferences

Chlorine at levels above 3.5 mg / L cause a yellow-brown color upon the addition of the Bromcresol Green-Methyl Red Indicator Powder Pillow. Residual chlorine interference with the indicator may be removed by adding a drop of 0.1 N Sodium Thiosulfate Standard Solution (Cat. No. (323-32).

Highly colored or turbid samples may mask the color change at the end point. Use a pH meter for these samples, titrating to pH 8.3 for phenolphthalein alkalinity and the appropriate pH (see *Table 2*) for total alkalinity.

Summary of Method

Alkalinity is expressed as P (phenolphthalein) alkalinity or as T (total) alkalinity. Both types are determined by titration with a Sulfuric Acid Standard Solution to an end point evidenced by the color change of an indicator solution or determined with a pH meter. The P alkalinity is determined by titration to a pH of 8.3 and registers the total hydroxide and one half the carbonate present. The T alkalinity is determined by titration to a pH of 4.5. The total alkalinity includes all carbonate, bicarbonate and hydroxide alkalinity. Alternatively, total alkalinity end points may be determined by using a pH meter and titrating to the specific pH required for the sample composition.

Alkalinity

Required Reagents

Description	Quantity Required Per Test	Unit	Cat. No.
Bromcresol Green-Methyl Red Indicator Powder Pillows	1 pillow	100 / pkg	943-99
Phenolphthalein Indicator Powder Pillows	1 pillow	100 / pkg	942-99
Sulfuric Acid Standard Solution, 0.020 N	varies	1 L	203-53

Required Apparatus

Buret Clamp, double	1	each	328-00
Buret, Class A, 25-mL	1	each	26365-40
Select one or more based on sample volume:			
Cylinder, graduated, 5-mL		each	508-37
Cylinder, graduated, 10-mL		each	508-38
Cylinder, graduated, 25-mL		each	508-40
Cylinder, graduated, 50-mL		each	508-41
Flask, Erlenmeyer, 250-mL	1	each	505-46
Funnel, Micro	1	each	25843-35
Support Stand	1	each	563-00

Required Standards

Alkalinity Standard Solution, Voluette® Ampules, 0.500 N, 10-mL		16 / pkg	14278-10
Buffer Powder Pillows, pH 4.5		25	895-68
Buffer Powder Pillows, pH 8.3		25	898-68
Water, deionized		4 L	272-56

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

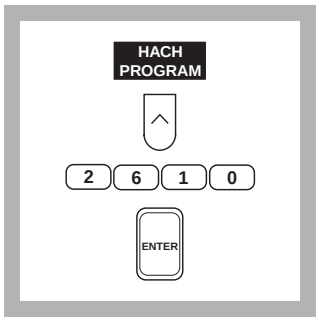
✔ **Method 8507** **Diazotization Method**

Powder Pillows or AccuVac® Ampuls **LR (0 to 0.300 mg/L NO_2^-–N)**

Scope and Application: *For water, wastewater and seawater; USEPA Approved* for wastewater analysis. The estimated detection limit for program numbers 2610 and 2620 are 0.0008 and 0.004 mg/L NO_2^-–N, respectively.*

* *Federal Register, 44(85),* 25505 (May 1, 1979)

Using Powder Pillows



1. Press the soft key under ***HACH PROGRAM***.

Select the stored program number for low range nitrite by pressing **2610** with the numeric keys.

Press: **ENTER**

Note: *If samples cannot be analyzed immediately, see Sample Collection, Storage and Preservation following these steps.*

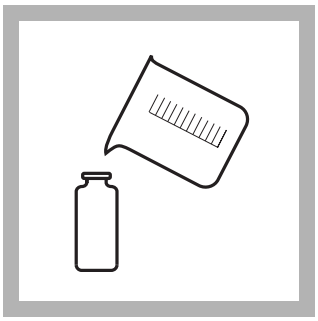
Note: *The Flow Cell and Sipper Modules can be used with this procedure. Use 25-mL samples and reagents with the Flow Cell Module.*

2. The display will show:

HACH PROGRAM: 2610 Nitrite, LR

The wavelength (λ), **507 nm**, is automatically selected.

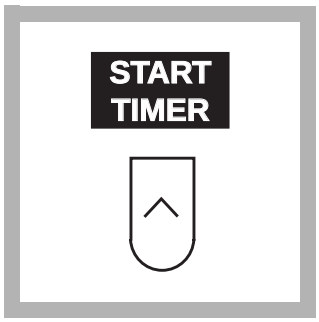
Note: *For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating steps 3 through 8, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under* ***ZERO****. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under* ***OPTIONS, (MORE)****, and then* ***BLANK:OFF****. Enter the reagent blank value and press* **ENTER***. Repeat for each new lot of reagent.*



3. Fill a sample cell with 10 mL of sample.

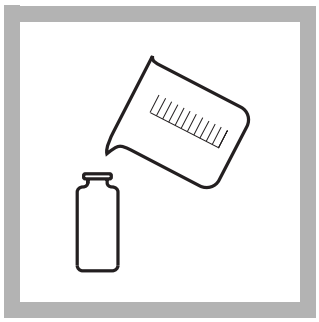
4. Add the contents of one NitraVer 3 Nitrate Reagent Powder Pillow (the prepared sample). Stopper. Shake to dissolve.

Note: *A pink color will develop if nitrite is present.*

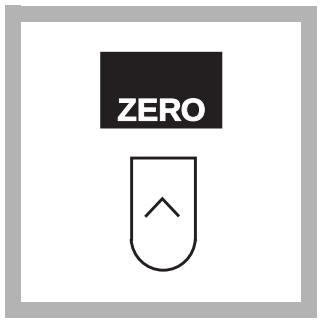


5. Press the soft key under ***START TIMER***.

A 20-minute reaction period will begin.



6. When the timer beeps, fill a second sample cell with 10 mL of sample (the blank). Place the blank into the cell holder.



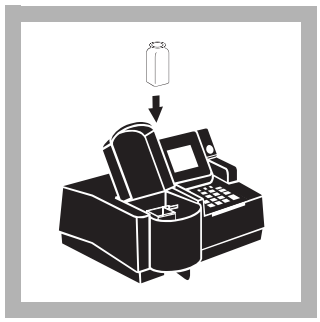
7. Press the soft key under ***ZERO***.

The display will show:

0.0000 mg/L NO_2^-–N

Note: *If you are using a reagent blank correction, the display will show the correction.*

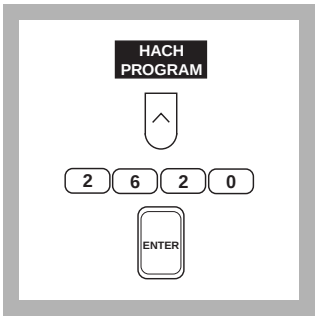
Note: *For alternate concentration units, press the soft key under* ***OPTIONS****. Then press the soft key under* ***UNITS*** *to scroll through the available options. Press* **ENTER** *to return to the read screen.*



8. Remove the stopper. Place the prepared sample into the cell holder. Close the light shield. Result in mg/L nitrite nitrogen (NO_2^-–N) will be displayed.

Note: *The result can be expressed as mg/L nitrite (NO_2^-). Press the soft keys under* ***OPTIONS*** *and then* ***FORM:*** *to scroll through the available options.*

Using AccuVac Ampuls



1. Press the soft key under ***HACH PROGRAM***.

Select the stored program number for low range nitrite by pressing **2620** with the numeric keys.

Press: **ENTER**

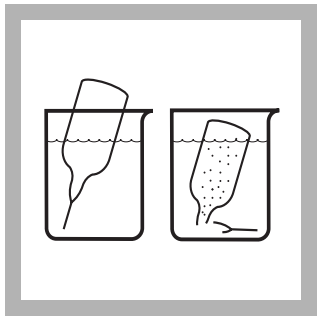
Note: *If samples cannot be analyzed immediately, see Sample Collection, Storage and Preservation following these steps.*

2. The display will show:

HACH PROGRAM: 2620
Nitrite, LR AV

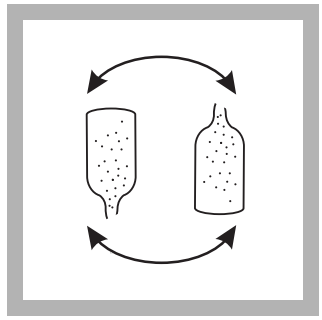
The wavelength (λ), **507 nm**, is automatically selected.

Note: *For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating steps 3 through 10, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under* ***ZERO****. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under* ***OPTIONS, (MORE)****, and then* ***BLANK:OFF****. Enter the reagent blank value and press* **ENTER***. Repeat for each new lot of reagent.*



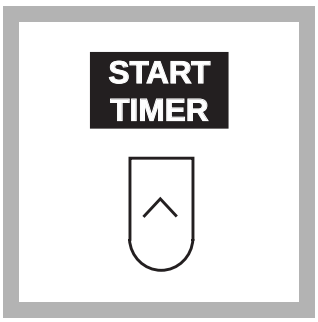
3. Collect at least 40 mL of sample in a 50-mL beaker. Fill a NitraVer 3 Nitrate AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills.*



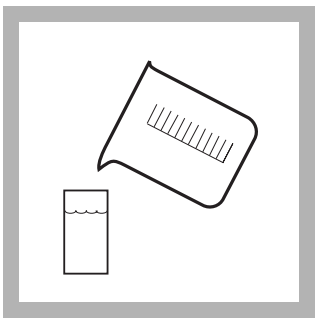
4. Invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will develop if nitrite is present.*



5. Press the soft key under ***START TIMER***.

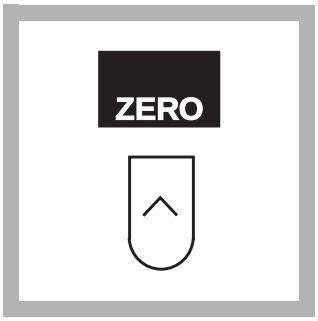
A 20-minute reaction period will begin.



6. When the timer beeps, fill a zeroing vial (the blank) with at least 10 mL of sample.

7. Insert the AccuVac Ampul Adapter into the sample cell module by sliding it under the thumb screw and into the alignment grooves. Fasten with the thumb screw.

8. Place the blank into the cell holder. Close the light shield.



9. Press the soft key under ***ZERO***.

The display will show:

0.0000 mg/L NO_2^-–N

Note: *If you are using a reagent blank correction, the display will show the correction.*

Note: *For alternate concentration units, press the soft key under* ***OPTIONS****. Then press the soft key under* ***UNITS*** *to scroll through the available options. Press* **ENTER** *to return to the read screen.*

10. Place the AccuVac Ampul into the cell holder. Close the light shield. Results in mg/L nitrate expressed as nitrogen (NO_2^-–N) will be displayed.

Note: *The results can be expressed as mg/L nitrate (NO_2^-). Press the soft keys under* ***METHOD OPTIONS****, then* ***FORM:*** *to scroll through the available options.*

Interferences

Table 1 Interfering Substances and Suggested Treatments

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

Sample Collection, Storage and Preservation

Collect samples in clean plastic or glass bottles. Store at 4 °C (30 °F) or lower if the sample is to be analyzed within 24 to 48 hours. Warm to room temperature before running the test.

Accuracy Check

Standard Solution Method
Preparing nitrite standards is difficult. A standard should be prepared by a trained chemist. Hach recommends using the standard preparation instructions in *Standard Methods for the Examination of Water and Wastewater,* Method 4500-NO_2^- B (p. 4–86 of 18th edition) Prepare a 0.150-mg/L standard.

Method Performance

Precision
Standard: 0.1500 mg/L NO_2^-–N

Program	95% Confidence Limits
2610	0.1494–0.1506 mg/L NO_2^-–N
2620	0.1496–0.1504 mg/L NO_2^-–N

For more information on determining precision data and method detection limits, refer to Section *1.5*.

Estimated Detection Limit

Program	EDL
2610	0.0008 mg/L NO_2^-–N
2620	0.0043 mg/L NO_2^-–N

For more information on derivation and use of Hach's estimated detection limit, see Section *1.5.2*. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, appendix B, see Section *1.5.1*.

Sensitivity
Program Number: 2610

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	0.00187 mg/L

Program Number: 2620

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	0.00203 mg/L

See Section *1.5.3 Sensitivity Explained* for more information.

Calibration Standard Preparation

Preparing nitrite standards is difficult. Calibration should be performed by a trained chemist. Hach recommends using the standard preparation instructions in *Standard Methods for the Examination of Water and Wastewater,* Method 4500-NO_2^- B (p. 4–86 of 18th edition).

Using the standards prepared above and the analysis procedure, generate a calibration curve.

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.

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. For additional information, refer to Section *1*.

Pollution Prevention and Waste Management

For information on pollution prevention and waste management, refer to Section *1*.

REQUIRED REAGENTS AND STANDARDS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
NitriVer 3 Nitrite Reagent Powder Pillows	1 pillow	100/pkg	21071-69

REQUIRED REAGENTS AND STANDARDS (Using AccuVac Ampuls)

NitriVer 3 Nitrite Reagent AccuVac Ampul	1 ampul	25/pkg	25120-25

REQUIRED EQUIPMENT AND SUPPLIES (Using Powder Pillows)

DR/4000 1-Inch Cell Adapter	1	each	48190-00
Sample Cells, matched pair, 1-inch, glass, with stoppers	2	pair	26126-02

REQUIRED EQUIPMENT AND SUPPLIES (Using AccuVac Ampuls)

Beaker, 50-mL	1	each	500-41
DR/4000 AccuVac Ampul Adapter	1	each	48187-00
Sample Cell, with cap (zeroing vial)	1	each	21228-00

OPTIONAL REAGENTS AND STANDARDS

Sodium Nitrite, ACS	454 g	2452-01
Water, deionized	4 liters	272-56

OPTIONAL EQUIPMENT AND SUPPLIES

Balance, analytical, 110 VAC	each	26103-00
Balance, analytical, 220 VAC	each	26103-02
DR/4000 Carousel Module Kit	each	48070-02
DR/4000 Flow Cell Module Kit, 1-inch	each	48070-04
DR/4000 Flow Cell Module Kit, 1-cm	each	48070-05
DR/4000 Sipper Module Kit, 1-inch	each	48090-03
Flask, volumetric, 1000-mL, Class B	each	547-53
Pipet, serological, 10-mL	each	532-38
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

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – **Call toll-free 800-227-4224**
Outside the U.S.A. – **Contact the HACH office or distributor serving you.**
On the Worldwide Web – **www.hach.com**; E-mail – **techhelp@hach.com**

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

16900-08

Digital Titrator

Model 16900

al/cth 8/97 23 ed
jnb/dk rev 4, 12/00

TRADEMARKS OF HACH COMPANY

AccuGrow®
AccuVac®
AccuVer™
AccuVial™
Add-A-Test™
AgriTrak™
AluVer®
AmVer™
APA 6000™
AquaChek™
AquaTrend®
BariVer®
BODTrak™
BoroTrace™
BoroVer®
C. Moore Green™
CA 610™
CalVer®
ChromaVer®
ColorQuik®
CoolTrak®
CuVer®
CyaniVer®
Digesdahl®
DithiVer®
Dr. F. Fluent™
Dr. H. Tueau™
DR/Check™
EC 310™
FerroMo®
FerroVer®
FerroZine®
FilterTrak™ 660
Formula 2533™
Formula 2589™
Gelex®
H_2O University™
H_2OU™
Hach Logo®
Hach One®
Hach Oval®
Hach.com™
HachLink™
Hawkeye The Hach Guy™
HexaVer®
HgEx™
HydraVer®
ICE-PIC™
IncuTrol®
Just Add Water™
LeadTrak®
M-ColiBlue24®
ManVer®
MolyVer®
Mug-O-Meter®
NetSketcher™
NitraVer®
NitriVer®
NTrak®
OASIS™
On Site Analysis.
Results You Can Trust℠
OptiQuant™
OriFlow™
OxyVer™
PathoScreen™
PbEx®
PermaChem®
PhosVer®
Pocket Colorimeter™
Pocket Pal™
Pocket Turbidimeter™
Pond In Pillow™
PourRite®
PrepTab™
ProNetic™
Pump Colorimeter™
QuanTab®
Rapid Liquid™
RapidSilver™
Ratio™
RoVer®
*sens**ion***™
Simply Accurate℠
SINGLET™
SofChek™
SoilSYS™
SP 510™
Spec√™
StablCal®
StannaVer®
SteriChek™
StillVer®
SulfaVer®
Surface Scatter®
TanniVer®
TenSette®
Test ‘N Tube™
TestYES!℠
TitraStir®
TitraVer®
ToxTrak™
UniVer®
VIScreen™
Voluette®
WasteAway™
ZincoVer®

TABLE OF CONTENTS

TABLE OF CONTENTS, continued

TABLE OF CONTENTS, continued

SPECIFICATIONS

Digital Titrator

Delivery: 800 digits/mL or 0.00125 mL/digit

Accuracy*: ± 1% for readings over 100 digits. (Uncertainty of readings is 1 digit. Most samples require more than 100 digits.)

Weight: 132 g (4.7 oz.)

Cartridges for the Digital Titrator

Volume: 13 mL

Number of tests: Most reagents are formulated to provide 100 typical titrations; the number may vary depending on sample concentration.

Weight (full): 56.75 g (2 oz.)

* Overall method accuracy includes, in addition to the Digital Titrator, other sources of error controlled by the analyst. The other sources of error include: sampling, sample volume, dilution (if required), end point detection, reagent quality, and interferences.

OPERATION

DANGER

Handling chemical samples, standards, and reagents can be dangerous. Review the necessary Material Safety Data Sheets and become familiar with all safety procedures before handling any chemicals.

DANGER

La manipulation des échantillons chimiques, étalons et réactifs peut être dangereuse. Lire les Fiches de Données de Sécurité des Produits (FDSP) et se familiariser avec toutes les procédures de sécurité avant de manipuler tous les produits chimiques.

PELIGRO

La manipulación de muestras químicas, estándares y reactivos puede ser peligrosa. Revise las fichas de seguridad de materiales y familiarícese con los procedimientos de seguridad antes de manipular productos químicos.

GEFAHR

Das Arbeiten mit chemischen Proben, Standards und Reagenzien ist mit Gefahren verbunden. Es wird dem Benutzer dieser Produkte empfohlen, sich vor der Arbeit mit sicheren Verfahrensweisen und dem richtigen Gebrauch der Chemikalien vertraut zu machen und alle entsprechenden Materialsicherheitsdatenblätter aufmerksam zu lesen.

PERIGO

A manipulação de amostras, padrões e reagentes químicos pode ser perigosa. Reveja a folha dos dados de segurança do material e familiarize-se com todos os procedimentos de segurança antes de manipular quaisquer produtos químicos.

GENERAL DESCRIPTION

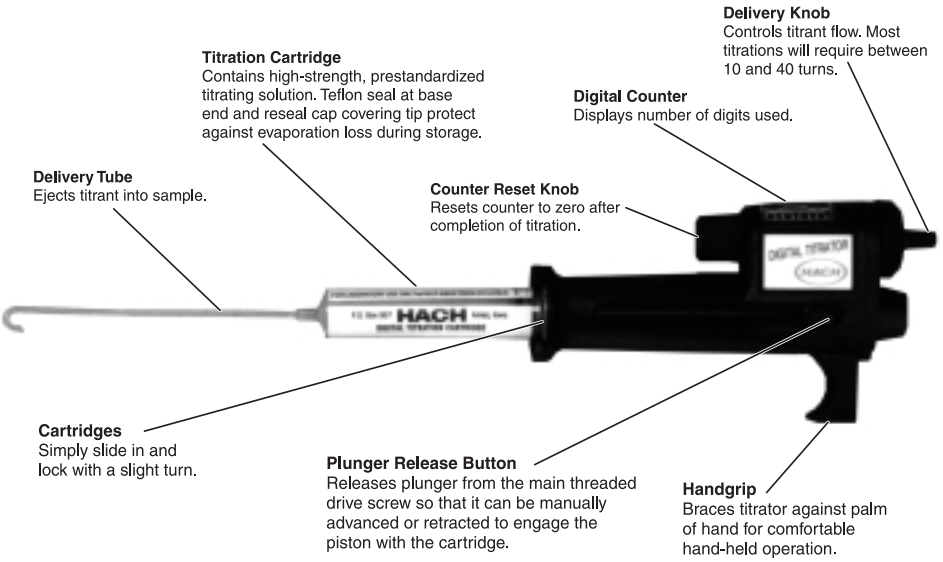
1.1 Introduction

Hach's Digital Titrator is a new concept in titrimetric analysis. It is a precision dispensing device fitted with compact cartridges that contain concentrated titrants. Accurate titrations are made without the bulk and fragility of conventional burets.

A main drive screw in the Digital Titrator controls a plunger which forces the concentrated titrant from a titration cartridge in a carefully regulated flow. The titrator body is constructed of precision-molded, heavy-duty, chemical- and impact-resistant acetal plastic. Accuracy is rated at ± 1% or better for a titration of more than 100 digits. For titrations less than 100, accuracy is ± 1 digit.

Titration solutions (titrants) are packaged in disposable polypropylene or Kynar® containers with Teflon-covered neoprene seals and polyethylene resealable closures to cover the cartridge tips. Each cartridge contains approximately 13 mL of titrating solution, enough for 50–100 average titrations. Titrant solutions are typically controlled to ± 0.5% concentration with normality and tolerances listed on the label. Titrant concentrations are designed for titrations of 10 to 40 turns (100 to 400 digits) of the delivery knob. For the most commonly used concentration ranges, the digits appearing in the counter window correspond to the sample concentration.

Figure 1 Hach Digital Titrator



Both portable and fixed-position titrations are possible with the Digital Titrator. The instrument has a grip for hand-held operation or it can be clamped to a TitraStir® Stir Plate or laboratory stand for stationary setups. See *Figure 1*.

Each Digital Titrator comes with five delivery tubes and a methods manual, which covers the most commonly tested parameters and the corresponding titrant cartridges. Right-angle (ninety-degree) delivery tubes for stationary setups are available as an optional accessory.

1.1.1 Following a Procedure for the First Time

Each method is divided into five sections: Procedure, Accuracy Check, Interferences, Summary of Method, and Reagents and Apparatus. For more information about how to select a procedure or for answers to chemical questions, see Hach's *Water Analysis Handbook* (literature 8376). For more information about chlorine measurement, also see the technical booklet titled, *Current Technology of Chlorine Analysis for Water and Wastewater* (literature 7019).

The **Procedure** details how to perform the method step-by-step. To select the appropriate sample volume and titration cartridge based on expected sample concentration, use the tables provided in each procedure. If the expected sample concentration is not known, start with one of the smaller sample volumes and determine its approximate concentration. Retest with the appropriate sample size.

The ranges in the table overlap to offer more flexibility. In most procedures, the number of digits used for each concentration range will be 100 to 400 digits.

To determine the actual concentration of the sample, use the correct digit multiplier for the sample volume and titration cartridge used.

Throughout the procedure, the notes will provide additional information.

The **Accuracy Check** provides a way to verify the results and determine if interferences are present. It also provides a method for checking the performance of reagents, the Digital Titrator and the operator's technique. Further information is provided in *Appendix A, Accuracy Check and Standard Additions*.

The **Interferences** section identifies common interferences causing inaccurate results and describes how to eliminate their effects. The interference levels are based on the sample volume that has 1.0 as the digit multiplier. Higher interference levels may be tolerated if a smaller sample is used.

The **Summary of Method** section discusses the chemical reaction taking place and information that applies to the entire procedure.

The **Reagents and Apparatus** list concludes the procedure. All the items required to perform the test are listed first and are available from Hach. The items listed in the notes or interferences sections are included in the optional listings.

1.2 Step-By-Step

1. Select a sample volume and titration cartridge corresponding to the expected sample concentration from the table given in each procedure.

   If the expected sample concentration is not known, start with one of the smaller sample volumes and determine its approximate concentration. Retest with the appropriate sample size.

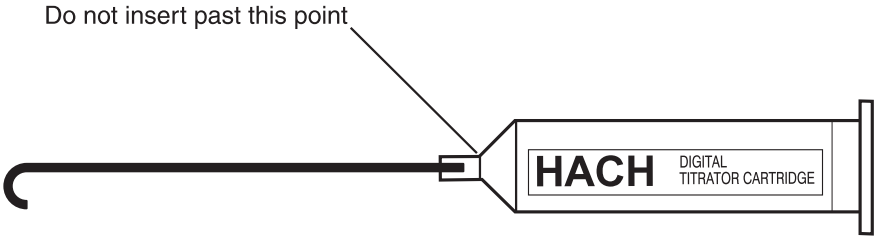
2. Slide the cartridge into the titrator receptacle and lock in position with a slight turn. See *Figure 2.*

Figure 2 Sliding the Cartridge into Place

3. Remove the polyethylene cap and insert a clean delivery tube into the end of the cartridge until it is tight. See *Figure 3.* Use a straight tube with a hook at the end for hand-held titrations; use a 90° tube with a hook at the end for stationary setups.

Do not insert tube past cartridge extension; see illustration below. In some instances, it might be necessary to remove a small burr on the leading edge of the tube before insertion.

Figure 3 Inserting the Delivery Tube



4. For stationary titrations, use a TitraStir Stir Plate or a clamp holder and clamp to attach the titrator to a laboratory stand. See *Figure 4* and *Figure 5*.

The TitraStir Stir Plate holds the Digital Titrator during the titration and also stirs the sample at a constant speed, leaving the analyst free to detect the end point. When a TitraStir Stir Plate is used, substitute or add the following Optional Apparatus.

APPARATUS

Description	Quantity Required Per Test	Unit	Cat. No.
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	1	5/pkg	41578-00
Flask, Erlenmeyer, 125 mL	1	each	505-43
Flask, Erlenmeyer, 250 mL	1	each	505-46
Stir Bar, 28.6 x 7.9 mm	1	each	20953-52
TitraStir® Stir Plate, 115 Vac	1	each	19400-00
TitraStir® Stir Plate, 230 Vac	1	each	19400-10

5. To start titrant flowing and flush the delivery tube, hold the tip of the cartridge up. Advance the plunger release button to engage the piston with the cartridge (push the button in and toward the cartridge). Do not expel solution when pushing the piston toward the cartridge. Turn the delivery knob until air is expelled and several drops of solution flow from the tip. As you turn the knob a drive screw pushes a piston against the cartridge seal and forces liquid out through the delivery tube. Then use the counter reset knob to turn the digital counter back to zero and wipe the tip. The tip can be rinsed with deionized water rather than wiped, if desired.

Figure 4 Using the TitraStir® Stir Plate

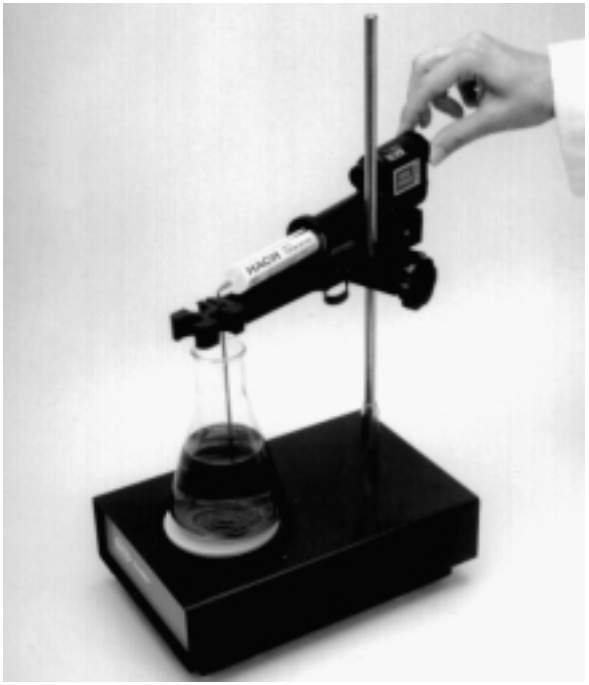


Figure 5 Using a Laboratory Stand

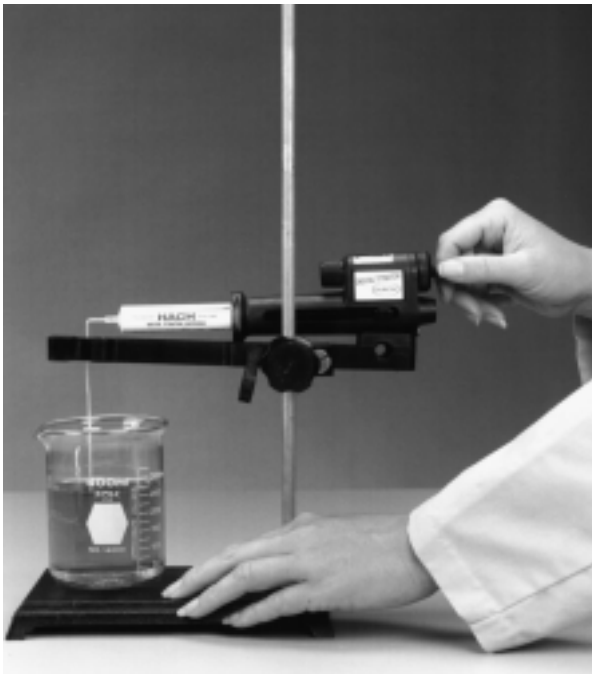


Figure 6 Titrating the Sample

6. Use the smallest appropriate graduated cylinder or pipet to measure the sample volume from the given table. Transfer the sample into a 125-mL or 250-mL Erlenmeyer flask. Dilute to the appropriate total volume with deionized water if necessary.

Note: *Sample volume measurements and dilutions (if required) must be made accurately. However, final total volume of titrated solution is not critical.*

7. Add the necessary reagents to the sample and swirl to mix.

8. Immerse the delivery tube tip in the solution and swirl the flask while titrating. Titrate by turning the delivery knob. Keep turning the knob and swirling the sample until the end point is reached. Record the number of digits that appear in the digital counter window. See *Figure 6*.

Note: *The number of digits required will usually range from 100 to 400. In nearly all of the procedures if the digits required is less than 100 or more than 400, an alternate sample volume or titrant cartridge should be used.*

Note: *Inaccurate results will occur if the delivery tube tip is held out of the solution rather than under the solution surface.*

9. Calculate the concentration of your sample by using the following formula:

Digits Required × Digit Multiplier = Sample Concentration

Where:

Digits Required = the number that appeared in the digital counter window in Step 8.

Digit Multiplier = the number from the table given in the procedure. It takes into account the sample dilution and titrant strength.

10. After completing testing for the day, press the plunger release button and manually retract the plunger into the body of the titrator. Remove the cartridge. Remove the delivery tube and reseal the cartridge with the polyethylene cap. See *Figure 7*.

Figure 7 Retracting the Plunger

11. Discard or clean the delivery tube immediately after use. To clean, force water, then air, into the tube opening with a syringe or wash bottle.

1.3 Helpful Hints

1.3.1 To Reuse a Partially Emptied Cartridge

1. With the plunger fully retracted, attach cartridge to the titrator.
2. Press the plunger release; then manually push the plunger against the cartridge seal.
3. Attach a delivery tube. Hold the tip of the cartridge up. Eject air and a few drops of titrant, zero the counter, and wipe the tip.
4. Titrate as usual.

1.3.2 To Calculate Titrant Volume Used

Normalities of many Hach titration cartridge solutions have been designed so that the number of digits used in a titration corresponds to the sample concentration in mg/L. To determine the volume used in mL, divide the Digital Titrator reading by 800.

1.3.3 To Fill Your Own Titration Cartridges

Cartridges may be cleaned and refilled, or new empty cartridges, Cat. No. 14495-01, can be purchased from Hach Company. See *Figure 8*. When preparing to refill old cartridges, push the cartridge seal out of the cartridge with air pressure applied through the tip. Cap the tip, fill with solution and reinsert the cartridge seal using care to avoid wrinkling the Teflon sheath. Filling also can be accomplished at the tip with a syringe.

Figure 8 Digital Titrator Cartridges

1.3.4 Verifying Technique

Whenever procedures are changed or new equipment is used, it is helpful to run a sample of known concentration. This technique will confirm the operator is following the procedure correctly and the new equipment is working properly. One objective important to Hach Company is making our tests self-verifying. This means Hach makes the tools available so the operator can check their own work for accurate results without relying on an outside lab or chemist.

For most of the tests in this manual, *Table 1* on page *20* lists each procedure, the suggested standard, the volume of standard needed, the titration cartridge used, and the number of expected digits when the test is performed correctly. The suggested standards are Voluette® or PourRite™ Ampules whenever possible because of their superior accuracy and stability.

To use titration standards follow these steps:

1. Select the procedure of interest and order the appropriate standard. Use the given catalog numbers.
2. Measure the volume of standard to be used as the sample in the procedure using a TenSette® Pipet or Class A pipet.
3. Perform the procedure as written, adding deionized water as necessary.
4. After titrating, the required number of digits should approximately equal the expected digits.

Call Hach Technical and Customer Service (1-800-227-4224) for additional help.

Table 1 Titration Standards

Procedure (Parameter)	Standard Description (Cat. No.)	Volume of Standard (mL)	Titration Cartridge (Cat. No.)	Expected Digits
Acid-Base: Acid	0.500 N H_2SO_4 (2121-26)	1.0	1.600 N NaOH (14379-01)	250
		5.0	8.00 N NaOH (14381-01)	250
Base	0.500 N Na_2CO_3 (14278-10)	1.0	1.600 N H_2SO_4 (14389-01)	250
		5.0	8.00 N H_2SO_4 (14391-01)	250
Acidity	0.500 N H_2SO_4 (2121-26)	0.1	0.1600 N NaOH (14377-01)	250
		1.0	1.600 N NaOH (14379-01)	250
Alkalinity	0.500 N Na_2CO_3 (14278-10)	0.1	0.1600 N H_2SO_4 (14388-01)	250
		1.0	1.600 N H_2SO_4 (14389-01)	250
Calcium*: mg/L $CaCO_3$	10,000 mg/L $CaCO_3$ (2187-10)	0.1	0.0800 M EDTA (14364-01)	100
		1.0	0.800 M EDTA (14399-01)	100
G.d.h.	10,000 mg/L $CaCO_3$ (2187-10)	0.2	0.1428 M EDTA (14960-01)	112
		1.0	0.714 M EDTA (14959-01)	112
Carbon Dioxide	10,000 mg/L CO_2 (14275-10)	0.2	0.3636 N NaOH (14378-01)	100
		2.0	3.636 N NaOH (14380-01)	
Chloride	12,500 mg/L Cl (14250-10)	0.1	0.2256 N $Hg(NO_3)_2$ (14393-01)	125
		0.1	0.2256 N $AgNO_3$ (14396-01)	125
		1.0	1.128 N $AgNO_3$ (14397-01)	250
		1.0	2.256 N $Hg(NO_3)_2$ (921-01)	125

Table 1 Titration Standards (Continued)

Procedure (Parameter)	Standard Description (Cat. No.)	Volume of Standard (mL)	Titration Cartridge (Cat. No.)	Expected Digits
Chlorine	~50 mg/L Cl_2 (14268-20) (see certificate)	2.0	0.02256 N $Na_2S_2O_3$ (24091-01)	varies**
	~25 mg/L Cl_2 (26300-20)	0.5	0.00564 N FEAS (22923-01)	varies***
Chromate	1000 mg/L Cr (2231 mg/L CrO_4) (14664-42)	1.0	0.2068 N $Na_2S_2O_3$ (22676-01)	223
Hardness: mg/L $CaCO_3$	10,000 mg/L $CaCO_3$ (2187-10)	0.1	0.0800 M EDTA (14364-01)	100
		0.1	0.0800 M CDTA (14402-01)	100
		1.0	0.800 M EDTA (14399-01)	100
		1.0	0.800 M CDTA (14403-01)	100
G.d.h.	10,000 mg/L $CaCO_3$ (2187-10)	0.2	0.1428 M EDTA (14960-01)	112
		1.0	0.714 M EDTA (14959-01)	112
Iron	50 mg/L Fe (14254-10)	10.0	0.0716 M TitraVer (20817-01)	200
	1000 mg/L Fe (2271-42)	10.0	0.716 M TitraVer (20818-01)	100
Oxygen, Dissolved****	10 mg/L as DO (401-11)	100	0.2000 N $Na_2S_2O_3$ (22675-01)	500
		200	2.00 N $Na_2S_2O_3$ (14401-01)	100
Sulfite	5000 mg/L SO_3 (22674-10)	1.0	0.3998 N KIO_3–KI (14961-01)	250

* One to two drops of Magnesium Standard Solution (10 g/L as $CaCO_3$) must be added to get a sharp end point. These added drops will not change the results.

** The expected digits equal the volume of standard times the concentration on the certificate (e.g., 2 mL x 50 mg/L = 100 digits).

*** The expected digits equals the volume of standard times the concentration on the certificate times the constant, 4. (Example: 0.5 mL x 50 mg/L x 4 = 100 digits)

**** Add one Sulfamic Acid Powder Pillow to the volume of standard and follow Steps 10 to 12 in the Dissolved Oxygen Procedure. It is not necessary to add the first two reagents.

1.4 Adapting a Buret Titration to the Digital Titrator

Adapt any standard titration procedure using a buret to the Digital Titrator by using the following procedure.

1. Determine the approximate number of digits required. The Digital Titrator dispenses 1 mL per 800 digits on the counter. Using the following equation, determine the digits required for your buret method.

$$\text{Digits Required} = \frac{N_t \times mL_t \times 800}{N_c}$$

Where:

N_t = Normality of buret titrant
mL_t = milliliters of buret titrant required for an average titration
N_c = Normality of Digital Titrator cartridge

2. If the number of digits required is within the range of 70 to 350, you can use the procedure as written, substituting the Digital Titrator directly for the buret. Or, if the number of digits is outside of this range, make the following modifications:

a. If the number of digits required is more than 350, reduce the sample size to save titrant.

b. If the number of digits required is less than 70, increase the sample size to increase precision.

c. If the sample size is altered, adjust the amount of buffering or indicating reagents by the same proportion.

3. When using the Digital Titrator for your buret method, note the number of digits required for a sample titration. To convert the digits required to the equivalent number of milliliters if the buret method was used, calculate:

$$\text{Equivalent Buret Milliliters} = \text{Digits Required} \times \frac{N_c}{800 \times N_t}$$

If the sample size was changed, adjust the equivalent buret milliliters accordingly. If the sample size was increased, reduce the equivalent buret milliliters; if the sample size was reduced increase the equivalent buret milliliters. Multiply the equivalent

buret milliliters by any normally used factors to calculate concentration in oz/gal, g/L, etc.

Example: Adapt a buret procedure, which normally requires about 20 mL of a 0.4 N titrant, to the Digital Titrator. Try an 8.0 N titration cartridge. The first equation above gives:

$$\text{Digits Required} = \frac{0.4 \times 20 \times 800}{8.0} = 800 \text{ digits}$$

Because this would use excessive titrant, reduce the sample size to one fourth its normal size to reduce the digits required to 200, well within the recommended range.

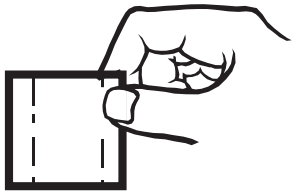
Upon completion of the titration using the smaller sample size, calculate the equivalent buret milliliters by the second equation above. If 205 were the digits required:

$$\text{Equivalent Buret Milliliters} = \frac{205 \times 8.0}{800 \times 0.4} = 5.13 \text{ mL}$$

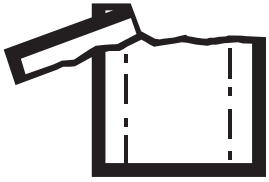
Multiply the 5.13 mL by 4 to account for the reduction in sample size to give the true equivalent buret milliliters of 20.5 mL. If the buret method called for multiplying the number of milliliters of titrant by a factor to calculate the concentration of a sample component, then multiply 20.5 by that factor.

1.5 Using PermaChem® Powder Pillows

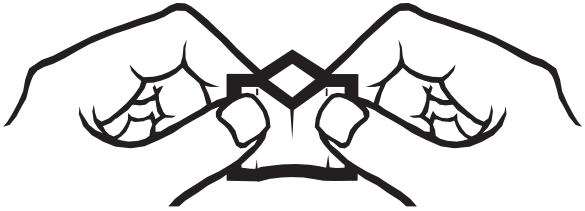
1. **Tap** the PermaChem on a hard surface to collect the powdered reagent in the bottom.



2. **Tear** across on the dotted pillow line marked “TEAR” holding the pillow away from your face.



3. Using two hands, **Push** both sides toward each other until thumbs and forefingers form a diamond. Make sure to **Crease** the foil pack, so that it forms a spout.



4. **Pour** the pillow contents into the sample. The polyfilm lining is specially formulated to deliver all the powder necessary for accurate results (no tapping on the vessel edge is necessary).

1.6 Safety

Safety is the responsibility of each individual when performing analysis procedures, and the analyst must develop and maintain good safety habits. Because many of the procedures in this methods handbook use potentially hazardous chemicals and apparatus, it is important that the analyst practice good laboratory techniques to minimize accidents. The following paragraphs present several techniques applicable to water analysis in the laboratory and in the field. They are not all inclusive, of course, nor do they apply only to the procedures provided in this handbook. They are general in nature but emphasize practices that are often key factors in personal injury incidents.

- Read labels carefully. Never remove the label from a reagent container. When preparing a reagent or standard solution, be sure to label the container clearly and date it.

- A Material Safety Data Sheet (MSDS) comes with each reagent. This sheet contains helpful information on first aid,

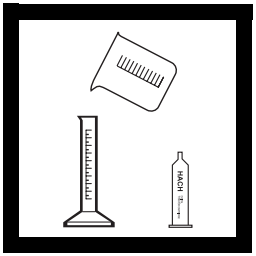
spill and disposal procedures, and precautionary measures and should be read before using the product.

- Warning labels also appear on some of the apparatus used with the test procedures.
- Wear protective clothing when handling chemicals that cause irritation or burns. Eye protection in particular is important to guard against spattering and splashes from accidental spills when caustic materials are being used.
- Use tongs or finger cots when transferring apparatus that is hot.
- Use mechanical pipetters: Mouth pipetting could result in accidentally ingesting dangerous chemicals. Make a habit of using mechanical pipet fillers for all pipetting. This will avoid mistakes that could cause serious injury.
- Use special care with dangerous chemicals and apparatus.
- Follow the test procedure steps carefully and observe all precautionary measures. It is good practice to read the entire procedure carefully before beginning the procedure. Use safety equipment, such as pipet fillers, protective clothing, and ventilating hoods, appropriate for the test being conducted. Wipe up all spills promptly. Do not smoke or eat in an area where toxic or irritating chemicals are used. Use reagents and apparatus only as they were meant to be used and use them only as directed in the test procedure. Do not use damaged labware and malfunctioning equipment.

TITRATION PROCEDURES

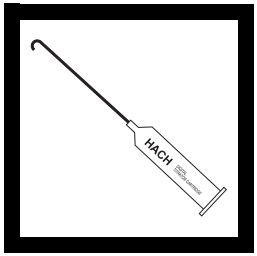
ACID-BASE (10 to 4000 mg/L as meq/L)

Acid Determination

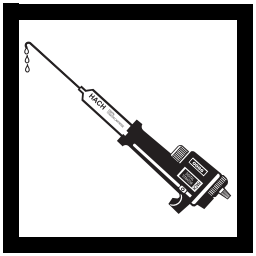


1. Select the sample volume corresponding to the expected acid concentration in milliequivalents (meq)/L or normality (N) from *Table 1*.

Note: *See Sampling and Storage following these steps.*



2. Insert a clean delivery tube into the appropriate Sodium Hydroxide Titration Cartridge. Attach the cartridge to the titrator body. See *General Description*, *Step-by-Step*, for assembly instructions.



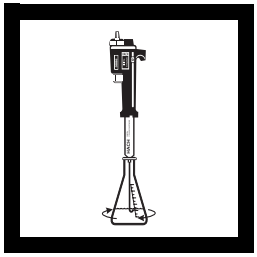
3. Flush the delivery tube by turning the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

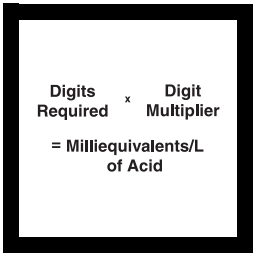
4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

5. Add the contents of one Phenolphthalein Indicator Powder Pillow and swirl to mix. The solution should be colorless.

Note: *Four drops of Phenolphthalein Indicator Solution may be substituted for the Phenolphthalein Indicator Powder Pillow.*



6. Place the delivery tube tip into the solution and swirl the flask while titrating with sodium hydroxide until a light pink color forms and persists for 30 seconds. Record the number of digits required.



7. Calculate:

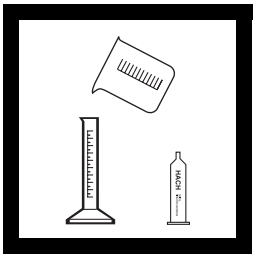
Digits Required x Digits Multiplier = Milliequivalents per Liter of Acid

Note: *To determine the normality of the sample, divide the milliequivalents per liter obtained by 1000.*

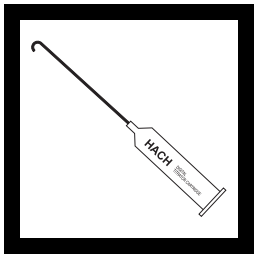
Table 1

Range meq/L	Range N	Sample Volume (mL)	Titration Cartridge	Catalog Number	Digit Multiplier
1-4	0.001-0.004	100	1.6 N NaOH 1.6 N H_2SO_4	14379-01 14389-01	0.02
4-10	0.004-0.01	50	1.6 N NaOH 1.6 N H_2SO_4	14379-01 14389-01	0.04
10-40	0.01-0.04	100	8 N NaOH 8 N H_2SO_4 8 N HCl	14381-01 14391-01 14390-01	0.1
20-80	0.02-0.08	50	8 N NaOH 8 N H_2SO_4 8 N HCl	14381-01 14391-01 14390-01	0.2
50-200	0.05-0.2	20	8 N NaOH 8 N H_2SO_4 8 N HCl	14381-01 14391-01 14390-01	0.5
100-400	0.1-0.4	10	8 N NaOH 8 N H_2SO_4 8 N HCl	14381-01 14391-01 14390-01	1.0
200-800	0.2-0.8	5	8 N NaOH 8 N H_2SO_4 8 N HCl	14381-01 14391-01 14390-01	2.0
500-2000	0.5-2	2	8 N NaOH 8 N H_2SO_4 8 N HCl	14381-01 14391-01 14390-01	5.0
1000-4000	1-4	1	8 N NaOH 8 N H_2SO_4 8 N HCl	14381-01 14391-01 14390-01	10.0

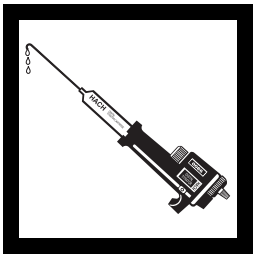
Base Determination



1. Select the sample volume corresponding to the expected base concentration in milliequivalents/L or normality from *Table 1*.



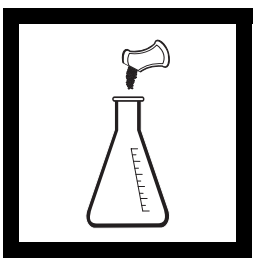
2. Insert a clean delivery tube into the appropriate Hydrochloric Acid or Sulfuric Acid Titration Cartridge. Attach the cartridge to the titrator body. See *General Description Section, Step-by-Step*, for assembly instructions, if necessary.



3. Flush the delivery tube by turning the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

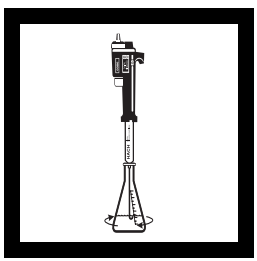
Note: *For added convenience use the TitraStir Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

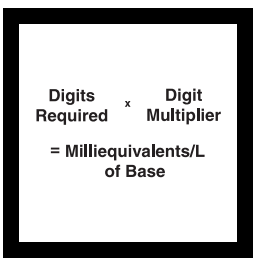


5. Add the contents of one Phenolphthalein Indicator Powder Pillow and swirl to mix. The solution should be a pink color.

Note: *Four drops of Phenolphthalein Indicator Solution may be substituted for the Phenolphthalein Indicator Powder Pillow.*



6. Titrate with 8.00 N hydrochloric acid or sulfuric acid until the solution is colorless. Record the number of digits required.



7. Calculate:

Digits Required x Digit Multiplier = Milliequivalents per Liter of Base

Note: *To determine the normality of the sample, divide the milliequivalents per liter obtained by 1000.*

Sampling and Storage

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Minimize agitation or prolonged exposure to air. Sample may be stored at least 24 hours by cooling to 4 °C (39 °F) or below if they cannot be analyzed immediately. Warm to room temperature before analyzing.

Accuracy Check

Using a clean Class A 20.00 mL pipet, transfer 20.00 mL 0.100 N NaOH Standard Solution (for base determination) or 20.00 mL 0.100 N Sulfuric Acid Standard Solution (for acid determination) to a clean 250-mL Erlenmeyer flask. Dilute to about 100 mL with deionized water.

Follow the procedure for base determination using 8.00 N HCl or H_2SO_4 Titration Cartridge or for acid determination using 8.00 N NaOH Titration Cartridge. About 200 digits of titrant should be required.

Interferences

Highly colored or turbid samples may mask the color change at the end point. Use a pH meter for these samples.

Summary of Method

A measured amount of sample is treated with a colorimetric indicator and then titrated with a strong acid or base. The amount of titrant used is directly proportional to the milliequivalents of acid or base in the sample. These titrations also can be performed using a pH meter instead of a colorimetric indicator. In this case, titrate to pH 7 or to the pH required.

REQUIRED REAGENTS

(varies with sample characteristics)

Description	Cat. No.
Acid Determination Reagent Set (about 100 tests)	
1-10 meq/L includes: (1) 942-99, (1) 14379-01	24459-00
10-4,000 meq/L includes: (1) 942-99, (1) 14381-01	24460-00

REQUIRED REAGENTS, continued

Description	Unit	Cat. No.
Hydrochloric Acid Titration Cartridge, 8.00 N	each	14390-01
Phenolphthalein Indicator Powder Pillows	100/pkg	942-99
Sodium Hydroxide Titration Cartridge, 1.600 N	each	14379-01
Sodium Hydroxide Titration Cartridge, 8.00 N	each	14381-01
Sulfuric Acid Titration Cartridge, 1.600 N	each	14389-01
Sulfuric Acid Titration Cartridge, 8.00 N	each	14391-01
Water, deionized	4 L	272-56

REQUIRED APPARATUS

Digital Titrator	each	16900-01
Flask, Erlenmeyer, 250-mL	each	505-46
Select one or more based on sample concentration:		
Cylinder, graduated, 5-mL	each	508-37
Cylinder, graduated, 10-mL	each	508-38
Cylinder, graduated, 25-mL	each	508-40
Cylinder, graduated, 50-mL	each	508-41
Cylinder, graduated, 100-mL	each	508-42

OPTIONAL REAGENTS

Phenolphthalein Indicator Solution, 5 g/L	100 mL*	162-32
Sodium Hydroxide Standard Solution, 0.100 N	1000 mL	191-53
Sulfuric Acid Standard Solution, 0.100 N	1000 mL*	202-53

OPTIONAL APPARATUS

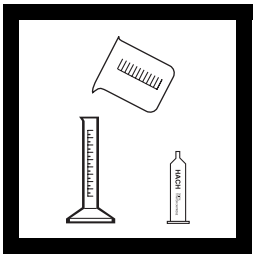
Bottle, wash, poly, 500-mL	each	620-11
Clamp, 2-prong, extension, 38-mm	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473-mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	5/pkg	41578-00
Pipet, volumetric, Class A, 1-mL	each	14515-35
Pipet, volumetric, Class A, 2-mL	each	14515-36
Pipet, volumetric, Class A, 5-mL	each	14515-37
Pipet, volumetric, Class A, 10-mL	each	14515-38
Pipet, volumetric, Class A, 20-mL	each	14515-20
Pipet, volumetric, Class A, 50-mL	each	14515-41
Pipet, volumetric, Class A, 100-mL	each	14515-42
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

* Contact Hach for larger sizes.

ACIDITY (10 to 4000 mg/L as $CaCO_3$)

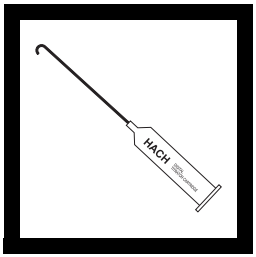
Methyl Orange and Phenolphthalein (Total) Methods

Methyl Orange Method — Method 8201

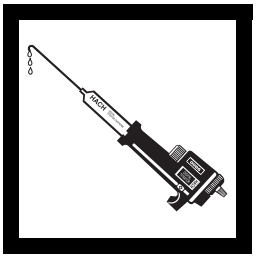


1. Select a sample volume and a Sodium Hydroxide (NaOH) Titration Cartridge corresponding to the expected acidity concentration as mg/L calcium carbonate ($CaCO_3$) from *Table 1*.

Note: *See Sampling and Storage following these steps.*

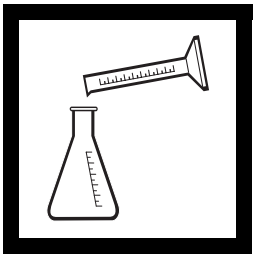


2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step* for assembly instructions, if necessary.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*



4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

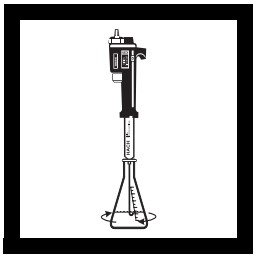
Note: *Minimize agitation because dissolved gases in the sample such as carbon dioxide, hydrogen sulfide and ammonia may be lost and cause inaccurate results.*

Table 1

Range (mg/L as $CaCO_3$)	Sample Volume (mL)	Titration Cartridge (N NaOH)	Catalog Number	Digit Multiplier
10-40	100	0.1600	14377-01	0.1
40-160	25	0.1600	14377-01	0.4
100-400	100	1.600	14379-01	1.0
200-800	50	1.600	14379-01	2.0
500-2000	20	1.600	14379-01	5.0
1000-4000	10	1.600	14379-01	10.0

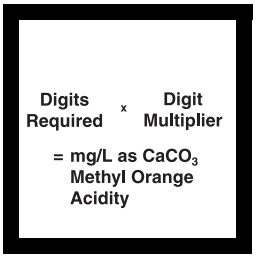
5. Add the contents of one Bromphenol Blue Indicator Powder Pillow and swirl to mix.

Note: *Six drops of Bromphenol Blue Indicator Solution may be substituted in this step.*



6. Place the delivery tube tip into the solution and swirl the flask while titrating with sodium hydroxide from yellow to blue-violet (pH 3.7). Record the number of digits required.

Note: *A solution of one pH 3.7 Buffer Powder Pillow and one Bromphenol Blue Indicator Powder Pillow in 50 mL of deionized water is recommended as a comparison for determining the proper end point color.*



7. Calculate:

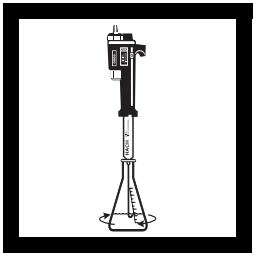
Digits Required x Digit Multiplier = mg/L as $CaCO_3$ Methyl Orange Acidity

Phenolphthalein (Total) Method **Method 8202**

1. Measure a second portion of the sample selected from *step 1 on page 35* into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

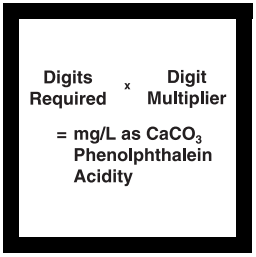
2. Add the contents of one Phenolphthalein Indicator Powder Pillow and swirl to mix.

Note: *Four drops of Phenolphthalein Indicator Solution may be substituted for the Phenolphthalein Indicator Powder Pillow.*



3. Titrate with sodium hydroxide from colorless to a light pink color that persists for 30 seconds. Record the number of digits required.

Note: *A solution of one pH 8.3 Buffer Powder Pillow and one Phenolphthalein Powder Pillow in 50 mL of deionized water is recommended as a comparison for determining the proper end point color.*



4. Calculate:

Digits Required x Digit Multiplier = mg/L as $CaCO_3$ Phenolphthalein Acidity

Sampling and Storage

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Minimize agitation or prolonged exposure to air. Samples may be stored at least 24 hours by cooling to 4 °C (39 °F) or below if they cannot be analyzed immediately. Warm to room temperature before analyzing.

Accuracy Check

Standard Additions Method

This accuracy check should be performed when interferences are suspected or to verify analytical technique.

1. Snap the neck off an Acidity Voluette® Ampule Standard, 0.500 N.

2. Use a TenSette® Pipet to add 0.1 mL of standard to the sample titrated in *step 6* for methyl orange acidity or *step 3* for phenolphthalein acidity. Resume titration back to the same end point. Note the number of digits required.

3. Repeat using two more additions of 0.1 mL. Titrate to the end point after each addition.

4. Each 0.1 mL addition of standard should require 25 additional digits of 1.600 N titrant or 250 digits of 0.1600 N titrant. If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

Interferences

- Highly colored or turbid samples may mask the color change at the end point. Use a pH meter for these samples.

- Chlorine may interfere with the indicators. Add one drop of 0.1 N Sodium Thiosulfate to eliminate this effect.

- To determine the phenolphthalein acidity of samples containing hydrolyzable metals such as iron, manganese or aluminum, use the following procedure:

  a. Adjust the sample in *step 1* for phenolphthalein acidity to pH 4.0 or less (if necessary) by using the Digital Titrator with an acid titration cartridge of identical normality to the Sodium Hydroxide Titration Cartridge used. Record the number of digits of acid added to lower the pH.

  b. Add five drops of 30% Hydrogen Peroxide Solution and boil the solution for 2-5 minutes.

  c. Cool to room temperature. Titrate following the Phenolphthalein Procedure *steps 2* and *3*. Subtract the number of digits of acid added to lower the pH from the number of digits required in *step 3* of the Phenolphthalein Procedure. Continue with *step 4*.

Summary of Method

Bromphenol blue (pH 3.7) or phenolphthalein (pH 8.3) indicator is used to titrate the sample with sodium hydroxide to a

colorimetric end point. Bromphenol blue gives a better end point than methyl orange indicator. Titration to pH 3.7 determines strong mineral acidity (also referred to as methyl orange acidity), whereas the pH 8.3 phenolphthalein end point includes weaker acid species as well, and represents the total acidity. The results are expressed in mg/L as calcium carbonate ($CaCO_3$) at a specified pH.

REQUIRED REAGENTS
(varies with sample characteristics)

Description	Unit	Cat. No.
Acidity Reagent Set (about 100 tests)		22728-00
Includes: (1) 942-99, (1) 14377-01, (1) 14379-01, (1) 14550-99		
Bromphenol Blue Powder Pillows	100/pkg	14550-99
Phenolphthalein Powder Pillows	100/pkg	942-99
Sodium Hydroxide Titration Cartridge, 0.1600 N	each	14377-01
Sodium Hydroxide Titration Cartridge, 1.600	each	14379-01
Water, deionized	4 L	272-56

REQUIRED APPARATUS

Digital Titrator	each	16900-01
Flask, Erlenmeyer, 250-mL	each	505-46
Select one or more based on sample concentration:		
Cylinder, graduated, 10-mL	each	508-38
Cylinder, graduated 25-mL	each	508-40
Cylinder, graduated 50-mL	each	508-41
Cylinder, graduated, 100-mL	each	508-42

OPTIONAL REAGENTS

Acidity Standard Solution, Voluette® Ampules, 0.500 N H_2SO_4, 10 mL	16/pkg	14330-10
Bromphenol Blue Indicator Solution	100 mL MDB	14552-32
Buffer Powder Pillows, pH 3.7	25/pkg	14551-68
Buffer Powder Pillows, pH 8.3	25/pkg	898-68
Hydrogen Peroxide Solution, 30%	200 mL MDB	144-45
Phenolphthalein Indicator Solution, 5 g/L	100 mL MDB*	162-32
Sodium Thiosulfate Standard Solution, 0.1 N	100 mL MDB*	323-32

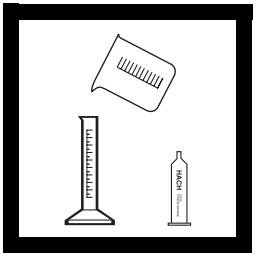
* Contact Hach for larger sizes.

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Bottle, wash, poly, 500-mL	each	620-11
Clamp, 2-prong extension, 38-mm	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473-mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	5/pkg	41578-00
Hot Plate, 3½-inch circular, 115 V	each	12067-01
Hot Plate, variable control, 4-inch circular, 230 V	each	12067-02
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, 10-mL	each	14515-38
Pipet, volumetric, Class A, 20-mL	each	14515-20
Pipet, volumetric, Class A, 25-mL	each	14515-40
Pipet, volumetric, Class A, 50-mL	each	14515-41
Pipet, volumetric, Class A, 100-mL	each	14515-42
Pipet Filler, safety bulb	each	14651-00
*sens**ion***™*1* Basic Portable pH Meter with electrode	each	51700-10
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10
Voluette® Ampule Breaker Kit	each	21968-0

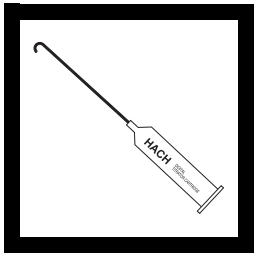
ALKALINITY (10 to 4000 mg/L as $CaCO_3$)

Phenolphthalein and Total Method

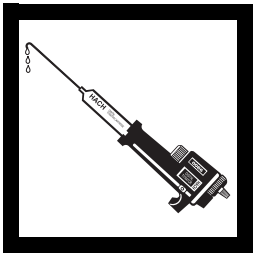


1. Select the sample volume and Sulfuric Acid (H_2SO_4) Titration Cartridge corresponding to the expected alkalinity concentration as mg/L calcium carbonate ($CaCO_3$) from *Table 1*.

Note: *See Sampling and Storage following these steps.*



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step* for assembly instructions, if necessary.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

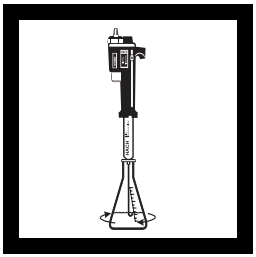
Table 1

Range (mg/L as $CaCO_3$)	Sample Volume (mL)	Titration Cartridge (H_2SO_4)	Catalog Number	Digit Multiplier
10-40	100	0.1600	14388-01	0.1
40-160	25	0.1600	14388-01	0.4
100-400	100	1.600	14389-01	1.0
200-800	50	1.600	14389-01	2.0
500-2000	20	1.600	14389-01	5.0
1000-4000	10	1.600	14389-01	10.0

5. Add the contents of one Phenolphthalein Indicator Powder Pillow and swirl to mix.

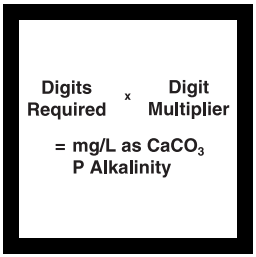
Note: *A solution of one pH 8.3 Buffer Powder Pillow and one Phenolphthalein Powder Pillow in 50 mL of deionized water is recommended as a comparison for determining the proper end point color.*

Note: *Four drops of Phenolphthalein Indicator Solution may be substituted for the Phenolphthalein Indicator Powder Pillow.*



6. If the solution turns pink, titrate to a colorless end point. Place the delivery tube tip into the solution and swirl the flask while titrating with sulfuric acid. Record the number of digits required.

Note: *If the solution is colorless before titrating with sulfuric acid, the Phenolphthalein (P) Alkalinity is zero; proceed with step 8.*



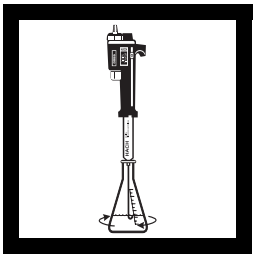
7. Calculate:

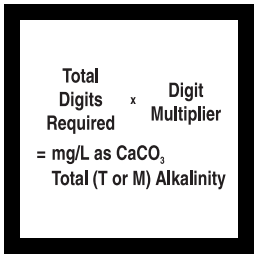
Digits Required x Digit Multiplier = mg/L $CaCO_3$ P Alkalinity

8. Add the contents of one Bromcresol Green-Methyl Red Indicator Powder Pillow to the flask and swirl to mix.

Note: *Four drops of Methyl Purple Indicator Solution may be substituted for the Bromcresol Green-Methyl Red Indicator Powder Pillow. Titrate from green to a gray end point (pH 5.1).*

Note: *Four drops of Bromcresol Green-Methyl Red Indicator Solution may be substituted for the Bromcresol Green-Methyl Red Indicator Powder Pillow.*





9. Continue the titration with sulfuric acid to a light greenish blue-gray (pH 5.1), a light violet-gray (pH 4.8), or a light pink (pH 4.5) color, as required by the sample composition; see *Table 2.* Record the number of digits required.

Note: *A solution of one Bromcresol Green-Methyl Red Powder Pillow and one pillow of the appropriate pH buffer in 50 mL of deionized water is recommended as a comparison for judging the proper end point color. If the pH 3.7 end point is used, use a Bromphenol Blue Powder Pillow instead of a Bromcresol Green-Methyl Red and titrate to a green end point.*

10. Calculate:

Total Digits Required x Digit Multiplier = mg/L as CaCO3 Total (T or M) Alkalinity

Note: *Carbonate, bicarbonate and hydroxide concentrations may be expressed individually using the relationships shown in Table 3.*

Note: *meq/L Alkalinity = mg/L as $CaCO_3$ ÷ 50.*

Table 2

Sample Composition	End Point
Alkalinity about 30 mg/L	pH 4.9
Alkalinity about 150 mg/L	pH 4.6
Alkalinity about 500 mg/L	pH 4.3
Silicates or Phosphates present	pH 4.5
Industrial waste or complex system	pH 4.5

Sampling and Storage

Collect samples in clean plastic or glass bottles. Fill completely and cap tightly. Avoid excessive agitation or prolonged exposure to air. Samples should be analyzed as soon as possible after collection but can be stored at least 24 hours by cooling to 4 °C (39 °F) or below. Warm to room temperature before analyzing.

Alkalinity Relationship Table

Total alkalinity primarily includes hydroxide, carbonate and bicarbonate alkalinities. The concentration of these alkalinities in a sample may be determined when the phenolphthalein and total alkalinities are known (see *Table 3*).

Table 3 Alkalinity Relationship

Row	Result of Titration	Hydroxide Alkalinity is equal to:	Carbonate Alkalinity is equal to:	Bicarbonate Alkalinity is equal to:
1	Phenolphthalein Alkalinity = 0	0	0	Total Alkalinity
2	Phenolphthalein Alkalinity equal to Total Alkalinity	Total Alkalinity	0	0
3	Phenolphthalein Alkalinity less than one half of Total Alkalinity	0	2 times the Phenolphthalein Alkalinity	Total Alkalinity minus two times Phenolphthalein Alkalinity
4	Phenolphthalein Alkalinity equal to one half of Total Alkalinity	0	Total Alkalinity	0
5	Phenolphthalein Alkalinity greater than one half of Total Alkalinity	2 times the Phenolphthalein minus Total Alkalinity	2 times the difference between Total and Phenolphthalein Alkalinity	0

To use the table follow these steps:

a. Does the phenolphthalein alkalinity equal zero? If yes, use Row 1.

b. Does the phenolphthalein alkalinity equal total alkalinity? If yes, use Row 2.

c. Multiply the phenolphthalein alkalinity by 2.

d. Select Row 3, 4, or 5 based on comparing the result of *step c* with the total alkalinity.

e. Perform the required calculations in the appropriate row, if any.

f. Check your results. The sum of the three alkalinity types will equal the total alkalinity.

For example:
A sample has 170 mg/L as $CaCO_3$ phenolphthalein alkalinity and 250 mg/L as $CaCO_3$ total alkalinity. What is the concentration of hydroxide, carbonate and bicarbonate alkalinities?

The phenolphthalein alkalinity does not equal 0 (it is 170 mg/L), see *step a*.

The phenolphthalein alkalinity does not equal total alkalinity (170 mg/L vs. 250 mg/L), see *step b*.

The phenolphthalein alkalinity multiplied by 2 = 340 mg/L, see *step c*.

Because 340 mg/L is greater than 250 mg/L, select Row 5, see *step d*.

The hydroxide alkalinity is equal to: (see *step e*).

340 – 250 = 90 mg/L hydroxide alkalinity

The carbonate alkalinity is equal to:

250 – 170 = 80
80 x 2 = 160 mg/L carbonate alkalinity

The bicarbonate alkalinity equals 0 mg/L.

Check: (see *step f*).

90 mg/L hydroxide alkalinity + 160 mg/L carbonate alkalinity +
0 mg/L bicarbonate alkalinity = 250 mg/L

The above answer is correct; the sum of each type equals the total alkalinity.

Accuracy Check

Standard Additions Method
This accuracy check should be performed when interferences are suspected or to verify analytical technique.

1. Snap the neck off an Alkalinity Standard Solution Voluette® Ampule, 0.500 N.
2. Use a TenSette® Pipet to add 0.1 mL of standard to the sample titrated in Steps 6 or 9. Resume titration back to the same end point. Record the number of digits needed.
3. Repeat, using two more additions of 0.1 mL. Titrate to the end point after each addition.
4. Each 0.1 mL addition of standard should require 25 additional digits of 1.600 N titrant or 250 digits of 0.1600 N titrant. If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

Interferences

- Highly colored or turbid samples may mask the color change at the end point. Use a pH meter for these samples.
- Chlorine may interfere with the indicators. Add one drop of 0.1 N Sodium Thiosulfate to eliminate this interference.

Summary of Method

The sample is titrated with sulfuric acid to a colorimetric end point corresponding to a specific pH. Phenolphthalein alkalinity is determined by titration to a pH of 8.3, as evidenced by the color change of phenolphthalein indicator, and indicates the total hydroxide and one half the carbonate present. M (methyl orange) or T (total) alkalinity is determined by titration to a pH between 3.7 and 5.1, and includes all carbonate, bicarbonate and hydroxide.

REQUIRED REAGENTS
(varies with sample characteristics)

Description	Unit	Cat. No
Alkalinity Reagent Set (about 100 tests)		22719-00
Includes: (1) 942-99, (1) 943-99, (1) 14388-01, (1) 14389-01		
Bromcresol Green-Methyl Red Powder Pillows	100/pkg	943-99
Phenolphthalein Powder Pillows	100/pkg	942-99
Sulfuric Acid Titration Cartridge, 1.600 N	each	14389-01
Sulfuric Acid Titration Cartridge, 0.1600 N	each	14388-01
Water, deionized	4 L	272-56

REQUIRED APPARATUS

Digital Titrator	each	16900-01
Flask, Erlenmeyer, 250-mL	each	505-46
Select one or more based on sample concentration:		
Cylinder, graduated, 10-mL	each	508-38
Cylinder, graduated, 25-mL	each	508-40
Cylinder, graduated, 50-mL	each	508-41
Cylinder, graduated, 100-mL	each	508-42

OPTIONAL REAGENTS

Alkalinity Standard Solution Voluette® Ampules, 0.500 N Na_2CO_3, 10-mL	16/pkg	14278-10
Bromcresol Green-Methyl Red Indicator Solution	100 mL MDB	23292-32
Bromphenol Blue Indicator Solution	100 mL MDB	14552-32
Bromphenol Blue Powder Pillows	100/pkg	14550-99
Buffer Powder Pillows, pH 3.7	25/pkg	14551-68
Buffer Powder Pillows, pH 4.5	25/pkg	895-68
Buffer Powder Pillows, pH 4.8	25/pkg	896-68
Buffer Powder Pillows, pH 5.1	25/pkg	897-68
Buffer Powder Pillows, pH 8.3	25/pkg	898-68
Methyl Purple Indicator Solution	100 mL MDB	21934-32
Phenolphthalein Indicator Solution, 5 g/L	100 mL MDB*	162-32
Sodium Thiosulfate Standard Solution, 0.1 N	100 mL MDB	323-32

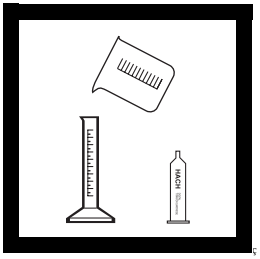
* Contact Hach for larger sizes.

OPTIONAL APPARATUS

Description	Unit	Cat. No
Bottle, wash, poly, 500-mL	each	620-11
Clamp, 2-prong extension, 38-mm	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473-mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	5/pkg	41578-00
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, 10-mL	each	14515-38
Pipet, volumetric, Class A, 20-mL	each	14515-20
Pipet, volumetric, Class A, 25-mL	each	14515-40
Pipet, volumetric, Class A, 50-mL	each	14515-41
Pipet, volumetric, Class A, 100-mL	each	14515-42
Pipet Filler, safety bulb	each	14651-00
*sens**ion**™1* Basic Portable pH Meter with electrode	each	51700-10
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10
Voluette® Ampule Breaker Kit	each	21968-00

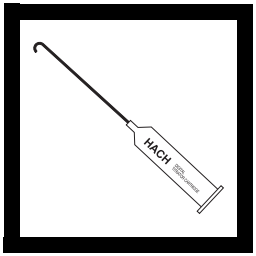
CARBON DIOXIDE (10 to 1000 mg/L as CO_2)

Using Sodium Hydroxide

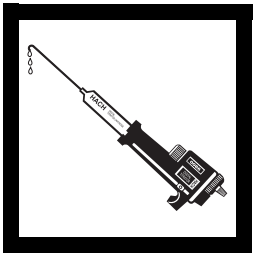


1. Select a sample size and a Sodium Hydroxide (NaOH) Titration Cartridge corresponding to the expected carbon dioxide (CO_2) concentration; see *Table 1*.

Note: *See Sampling and Storage following these steps.*



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step* for assembly instructions if necessary.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Collect a water sample directly into the titration flask by filling to the appropriate mark.

Note: *Minimize agitation because carbon dioxide may be lost.*

Note: *For most accurate results, check the calibration of the Erlenmeyer flask by measuring the proper volume in a graduated cylinder. Mark the proper volume on the flask with a permanent marker.*

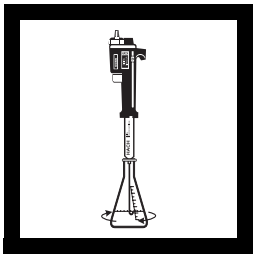
Table 1

Range (mg/L as CO_2)	Sample Volume (mL)	Titration Cartridge (N NaOH)	Catalog Number	Digit Multiplier
10-50	200	0.3636	14378-01	0.1
20-100	100	0.3636	14378-01	0.2
100-400	200	3.636	14380-01	1.0
200-1000	100	3.636	14380-01	2.0

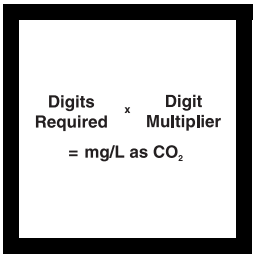
5. Add the contents of one Phenolphthalein Indicator Powder Pillow and mix.

Note: *Four drops of Phenolphthalein Indicator Solution may be substituted for the Phenolphthalein Indicator Powder Pillow.*

Note: *If a pink color forms, no carbon dioxide is present.*



6. Place the delivery tube tip into the solution and swirl the flask gently while titrating with sodium hydroxide from colorless to a light pink color that persists for 30 seconds. Record the number of digits required.



7. Calculate:

Total Digits Required x Digit Multiplier = mg/L as CO2

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 as soon as possible after collection. If immediate analysis is not possible, the samples may be stored for at least 24 hours by cooling to 4 °C (39 °F) or below. Before analysis, warm the samples to room temperature.

Accuracy Check

Standard Additions Method

This accuracy check should be performed when interferences are suspected or to verify analytical technique.

1. Snap the neck off a Carbon Dioxide Standard Solution Voluette® Ampule, 10,000 mg/L CO_2.

2. Use a TenSette® Pipet to add 0.1 mL of standard to the sample titrated in *step 6*. If using 0.3636 N titrant, use 1.0 mL of standard. Resume titration back to the same end point. Record the number of digits required.

3. Repeat, using additions of 0.2 mL and 0.3 mL (2.0 and 3.0). Titrate to the same end point after each addition.

4. Each addition of standard should require 50 additional digits of titrant. If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

Interferences

- Other acid components in the sample will be titrated and interfere directly in this determination.

- Sodium hydroxide standard solutions tend to lose strength slowly with age and should be checked periodically by titrating a known standard. Check the solution frequently (monthly) by titrating 50 mL of Potassium Acid Phthalate Standard Solution, 100 mg/L CO_2, using Phenolphthalein Indicator Solution. The titration should require 5.00 mL of titrant. If the volume required for this titration is greater than 5.25 mL, discard the sodium hydroxide and replace it with a fresh supply.

Summary of Method

Acidity due to carbon dioxide in a sample is titrated with sodium hydroxide to a phenolphthalein end point. Strong acids are assumed to be absent or of insignificant concentration. Request Hach's *Water Analysis Handbook,* Publication 8376, to obtain additional information on carbon dioxide determinations.

REQUIRED REAGENTS
(varies with sample characteristics)

Description	Unit	Cat. No.
Carbon Dioxide Reagent Set (about 100 tests)		22727-00
Includes: (1) 942-99, (1) 14378-01, (1) 14380-01		
Phenolphthalein Powder Pillows	100/pkg	942-99
Sodium Hydroxide Titration Cartridge, 0.3636 N	each	14378-01
Sodium Hydroxide Titration Cartridge, 3.636 N	each	14380-01
Water, deionized	4 L	272-56

REQUIRED APPARATUS

Digital Titrator	each	16900-01
Select one or more based on sample concentration:		
Flask, Erlenmeyer, 250 mL	each	505-46
Flask, Erlenmeyer, 125 mL	each	505-43

OPTIONAL REAGENTS

Carbon Dioxide Standard Solution Voluette® Ampules, 10,000 mg/L as CO_2, 10 mL	16/pkg	14275-10
Phenolphthalein Indicator Solution, 5 g/L	100 mL MDB*	162-32
Potassium Acid Phthalate Standard Solution, 100 mg/L as CO_2	100 mL	2261-42
Potassium Acid Phthalate Standard Solution, 400 mg/L as CO_2	500 mL	1885-49

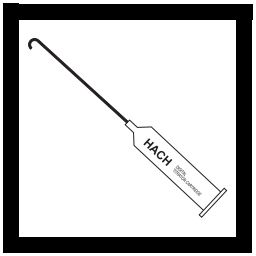
OPTIONAL APPARATUS

Clamp, 2-prong extension, 38 mm	each	21145-00
Clamp Holder	each	326-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	5/pkg	41578-00
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette® Pipet	50/pkg	21856-96
Pipet Filler, safety bulb	each	14651-00
*sens**ion**™1* Basic Portable pH Meter with electrode	each	51700-10
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10
Voluette® Ampule Breaker Kit	each	21968-00

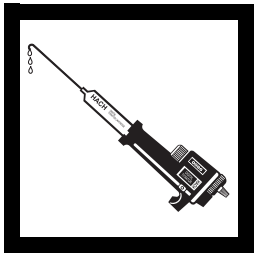
* Contact Hach for larger sizes.

CHELANT, FREE (0 to 20.0 mg/L as $CaCO_3$)

Using Magnesium Chloride



1. Insert a clean delivery tube into the Magnesium Chloride Titration Cartridge. Attach the cartridge to the titrator body. See *General Description*, *Step-By-Step*, for assembly instructions.



2. Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob until a few drops of titrant are expelled. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

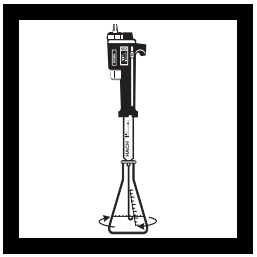
3. Use a graduated cylinder to measure the 100 mL of sample into a 125-mL Erlenmeyer flask.

Note: *Filter sample if necessary. If sample is boiler water or highly alkaline, refer to Interferences following these steps.*

4. Using the 1-mL calibrated dropper, add 2 mL of Hardness 1 Buffer Solution to the flask and swirl to mix.

5. Add the contents of one ManVer® 2 Hardness Indicator Powder Pillow to the flask and swirl to mix. If the solution turns blue, free chelant is present. Proceed to *step 6*. If the solution turns red, a deficiency of chelant exists.

Note: *Four drops of ManVer Hardness Indicator Solution or a 0.1 g scoop of ManVer 2 Hardness Indicator Powder may be substituted in this step.*



6. Place the delivery tube tip into the solution. While swirling the flask, titrate until a red-violet color appears. Record the number of digits required.

7. Calculate:

Digits Required x 0.10 = mg/L Free Chelant (as $CaCO_3$)

Note: *The results may be expressed as mg/L tetra-sodium EDTA (digits required x 0.38 = mg/L as Na_4 EDTA).*

Accuracy Check

Standard Additions Method

This accuracy check should be performed when interferences are suspected or to verify analytical technique.

1. Use a TenSette® Pipet to add 0.4 mL of 0.035 N EDTA Standard Solution to the solution titrated in *step 6*. Resume titration back to the same end point. Record the number of digits required.

2. Each 0.4 mL addition of standard should require 70 additional digits of 0.0800 M titrant. If this increase does not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

Interferences

- If chelant residual in boiler water is being analyzed, adjust the pH before adding the Hardness 1 Buffer Solution as follows:

  a. To another 100-mL sample, add 2 drops of Phenolphthalein Indicator Solution.

  b. Counting the drops, add 5.25 N Sulfuric Acid Standard Solution one drop at a time until the solution changes from pink to colorless. Discard this sample.

  c. To the actual 100-mL sample, add the same number of drops of 5.25 N Sulfuric Acid Standard Solution before adding the buffer in *step 4*.

- Orthophosphate causes a slow end point. Polyphosphate must be absent for accurate results.

- All apparatus must be scrupulously clean and rinsed frequently with acid and deionized water to remove any hardness present on the plastic or glass.

- Run reagent blanks occasionally, using deionized or distilled water in place of the sample. Subtract the value of the blank from the sample value before recording the final answer.

Summary of Method

Chelant residual is determined by titration with a standard solution of magnesium chloride at pH 10. The end point is determined by a color change from blue to red-violet.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Hardness 1 Buffer Solution	100 mL MDB	424-32
ManVer® 2 Hardness Indicator Powder Pillows	100/pkg	851-99
Magnesium Chloride Titration Cartridge, 0.0800 M	each	20625-01

REQUIRED APPARATUS

Cylinder, graduated, 100-mL	each	508-42
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 125 mL	each	505-43

OPTIONAL REAGENTS

EDTA Standard Solution, 0.035 N	100 mL MDB	23499-32
ManVer® 2 Hardness Indicator Powder	113 g	280-14
ManVer® 2 Hardness Indicator Solution	100 mL MDB*	425-32
Phenolphthalein Indicator Solution, 5 g/L	100 mL MDB*	162-32
Sulfuric Acid Standard Solution, 5.25 N	100 mL MDB	2449-32
Water, deionized	4 L	272-56

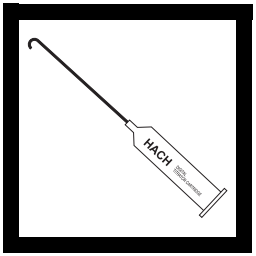
OPTIONAL APPARATUS

Clamp, 2-prong extension, 38 mm	each	21145-00
Clamp Holder	each	326-00
Clippers (shears), 7.25 inch	each	23694-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	5/pkg	41578-00
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, Erlenmeyer, 250 mL	each	505-46
Funnel, analytical, poly, 65 mm	each	1083-67
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette® Pipet	50/pkg	21856-96
Spoon, measuring, 0.5 gram	each	907-00
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

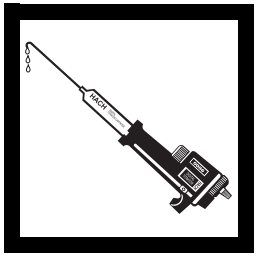
* Contact Hach for larger sizes.

CHELANT, TOTAL (0 to 40.0 mg/L as Na_4EDTA)

Using Bismuth Nitrate

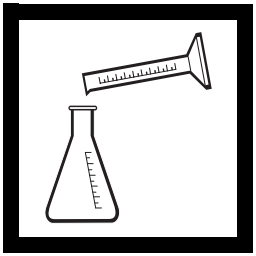


1. Insert a clean delivery tube into the Bismuth Nitrate Titration Cartridge. Attach the cartridge to the titrator body. See *General Description, Step-By-Step,* for assembly instructions.



2. Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob until a few drops of titrant are expelled. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*



3. Use a graduated cylinder to measure the 50 mL of clear sample into a 125-mL Erlenmeyer flask.

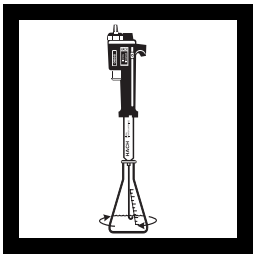
Note: *Filtration is required for turbid samples.*

4. Add the contents of one Ascorbic Acid Powder Pillow to the flask and swirl to mix.

5. Add the contents of one Methylthymol Blue Powder Pillow to the flask and swirl to mix.

6. If the solution in the flask is yellow, add one drop of 5.25 N Sulfuric Acid Standard Solution.

If the solution is blue, add 5.25 N Sulfuric Acid Standard Solution dropwise until the solution changes to yellow. Add one additional drop.



7. Place the delivery tube tip into the solution. While swirling the flask, titrate with the Bismuth Nitrate until the color changes from yellow to blue-green. Record the number of digits required.

Note: *Titrate slowly as the end point is approached.*

Note: *For best results, determine a reagent blank. Use 50 mL of deionized water in step 3. Subtract the number of digits required for the reagent blank from the number of digits required for titrating the sample.*

8. Calculate the final concentration:

Digits Required x 0.095 = Total Chelant (as mg/L Na_4EDTA)

Interferences

Interference from ferric iron (Fe^{3+}) is minimized by adding ascorbic acid. The end point should by approached slowly in samples containing ferric iron because the iron decreases the sharpness of the color change.

Summary of Method

Total chelant is determined by titrating an acid sample with bismuth nitrate the presence of methylthymol blue indicator. The end point is signaled by a color change from yellow to blue-green.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Ascorbic Acid Powder Pillows	100/pkg	14577-99
Bismuth Nitrate Titration Cartridge, 0.0200 M	each	24345-01
Methylthymol Blue Indicator Powder Pillows	50/pkg	22847-99
Sulfuric Acid Standard Solution, 5.25 N	100 mL MDB	2449-32

REQUIRED APPARATUS

Cylinder, graduated, poly, 100 mL	each	1081-42
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	5/pkg	41578-00
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 125 mL	each	505-43
Stir Bar, analytical, Teflon-coated, 50 mm	each	20953-55
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

OPTIONAL REAGENTS

Water, deionized	4 L	272-56

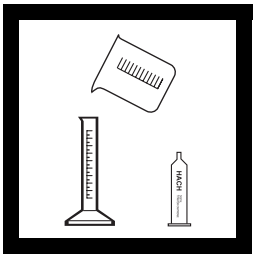
OPTIONAL APPARATUS

Clamp, 2-prong extension, 38 mm	each	21145-00
Clamp Holder	each	326-00
Clippers (shears), 7.25 inch	each	23694-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Filter paper, folded, 12.5 cm	100/pkg	1894-57
Flask, Erlenmeyer, 250 mL	each	505-46
Funnel, analytical, poly, 65 mm	each	1083-67

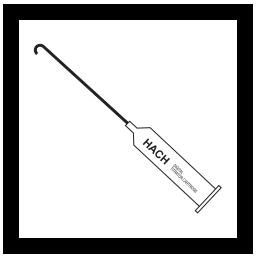
CHLORIDE

Mercuric Nitrate and Silver Nitrate Methods

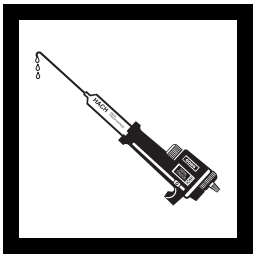
Mercuric Nitrate Method (10 to 8000 mg/L as Cl^-) Method 8206



1. Select the sample volume and Mercuric Nitrate Titration Cartridge corresponding to the expected chloride concentration from *Table 1*.



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description Section, Step-by-Step,* for assembly instructions if necessary.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, step 3 in Step-by-Step.*

4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

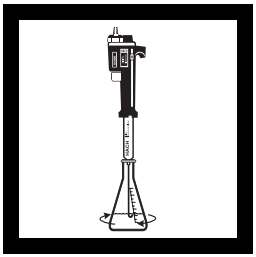
Note: *See following these steps.*

Table 1

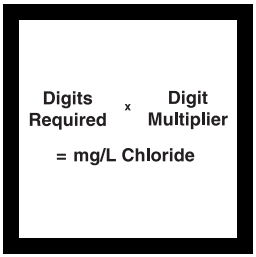
Range (mg/L as Cl^-)	Sample Volume (mL)	Titration Cartridge (N $Hg(NO_3)_2$)	Catalog Number	Digit Multiplier
10-40	100	0.2256	14393-01	0.1
40-160	25	0.2256	14393-01	0.4
100-400	100	2.256	921-01	1.0
200-800	50	2.256	921-01	2.0
500-2000	20	2.256	921-01	5.0
1000-4000	10	2.256	921-01	10.0
2000-8000	5	2.256	921-01	20.00

5. Add the contents of one Diphenylcarbazone Powder Pillow and swirl to mix.

Note: *Results will still be accurate if a small amount of the powder does not dissolve.*



6. Place the delivery tube tip into the solution and swirl the flask while titrating with mercuric nitrate from a yellow to light pink color. Record the number of digits required.



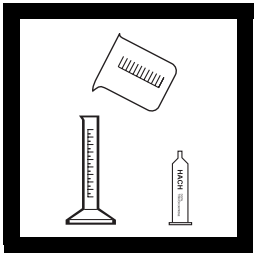
7. Calculate:

Digits Required x Digit Multiplier = mg/L Chloride

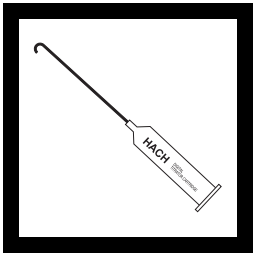
Note: *Results may be expressed as mg/L sodium chloride by multiplying the mg/L chloride by 1.65.*

Note: *meq/L Chloride = mg/L Cl^- ÷ 35.45.*

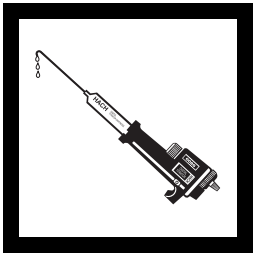
Silver Nitrate Method (10 to 10000 mg/L as Cl^-) Method 8207



1. Select the sample volume and Silver Nitrate Titration Cartridge corresponding to the expected chloride concentration from *Table 2*.



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description Section, Step-by-Step,* for assembly instructions if necessary.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Use a graduated cylinder or pipet to measure the sample volume from *Table 2*. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

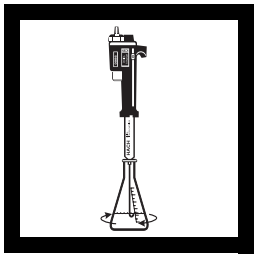
Note: *See following these steps.*

Table 2

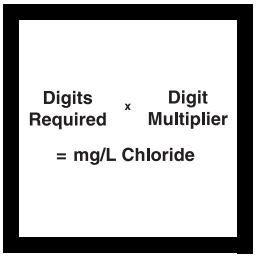
Range (mg/L as Cl^-)	Sample Volume (mL)	Titration Cartridge (N $AgNO_3$)	Catalog Number	Digit Multiplier
10-40	100	0.2256	14396-01	0.1
25-100	40	0.2256	14396-01	0.25
100-400	50	1.128	14397-01	1.0
250-1000	20	1.128	14397-01	2.5
1000-4000	5	1.128	14397-01	10.0
2500-10000	2	1.128	14397-01	25.0

5. Add the contents of one Chloride 2 Indicator Powder Pillow and swirl to mix.

Note: *Results will still be accurate if a small amount of the powder does not dissolve.*



6. Place the delivery tube tip into the solution and swirl the flask while titrating with silver nitrate from a yellow to red-brown color. Record the number of digits required.



7. Calculate:

Digits Required x Digit Multiplier = mg/L Chloride

Note: *Results may be expressed as mg/L sodium chloride by multiplying the mg/L chloride by 1.65.*

Note: *meq/L Chloride = mg/L Cl^- ÷ 35.45.*

Sampling and Storage

Collect at least 100 to 200 mL of sample in a clean glass or polyethylene container. Samples may be stored up to 7 days before analysis.

Accuracy Check

Standard Additions Method
This accuracy check should be performed when interferences are suspected or to verify analytical technique.

1. Snap the neck off a Chloride Standard Solution Voluette® Ampule, 12,500 mg/L Cl^-.

2. Use a TenSette® Pipet to add 0.1 mL of standard to the sample after titration in *step 6*. Resume titration back to the same end point. Record the number of digits required.

3. Repeat, using additions of 0.2 and 0.3 mL. Titrate to the end point after each addition.

4. Each 0.1 mL addition of standard should require 12.5 additional digits of 2.256 N titrant, 25 digits of 1.128 N titrant, or 125 digits of 0.2256 N titrant. If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions.*

Interferences Using the Mercuric Nitrate Method

- Chromate, ferric iron, and sulfite in excess of 10 mg/L interfere with this method.

- Eliminate sulfite interference by adding three drops of hydrogen peroxide, 30%, in *step 4*.

- Remove sulfide interference by adding the contents of one Sulfide Inhibitor Reagent Powder Pillow to about 125 mL of sample, mixing for one minute, and filtering through a folded filter paper.

- Iodide and bromide interfere directly and titrate as chloride.

- Neutralize strongly alkaline or acid samples to a pH of 2 to 7 with 5.25 N Sulfuric Acid Standard Solution or 5.0 N Sodium Hydroxide Standard Solution. Determine the amount of acid or base necessary in a separate sample because pH electrodes will introduce chloride into the sample.

Interferences Using the Silver Nitrate Method

- Iron in excess of 10 mg/L masks the end point.
- Orthophosphate in excess of 25 mg/L will precipitate the silver.
- Sulfite in excess of 10 mg/L interferes. Eliminate sulfite interference by adding three drops of 30% hydrogen peroxide in *step 4*.
- Remove sulfide interference by adding the contents of one Sulfide Inhibitor Reagent Powder Pillow to about 125 mL of sample, mixing for one minute, and filtering through a folded filter paper.
- Cyanide, iodide, and bromide interfere directly and titrate as chloride.
- Neutralize strongly alkaline or acid samples to a pH of 2 to 7 with 5.25 N Sulfuric Acid Standard Solution or 5.0 N Sodium Hydroxide Standard Solution. Determine the amount of acid or base necessary in a separate sample because pH electrodes will introduce chloride into the sample.

Summary of the Mercuric Nitrate Method

When using Mercuric Nitrate Standard Solution, the sample is titrated under acid conditions in the presence of diphenylcarbazone indicator. Upon addition of a slight excess of mercuric ion, a pink-purple complex is formed with the indicator, signaling the end point.

Summary of the Silver Nitrate Method

The sample is titrated with Silver Nitrate Standard Solution in the presence of potassium chromate (from the Chloride 2 Indicator Powder). The silver nitrate reacts with the chloride present to produce insoluble white silver chloride. After all the chloride has been precipitated, the silver ions react with the excess chromate present to form a red-brown silver chromate precipitate, marking the end point of the titration.

Request Hach's *Water Analysis Handbook*, Publication 8376, to obtain additional information on chloride determinations.

REQUIRED REAGENTS FOR THE MERCURIC NITRATE METHOD

Description	Unit	Cat. No.
Mercuric Nitrate Chloride Reagent Set (about 100 tests) Includes: (2) 836-46, (1) 921-01, (1) 14393-01		22726-00
Diphenylcarbazone Reagent Powder Pillows	100/pkg	836-99
Mercuric Nitrate Titration Cartridge, 0.2256 N	each	14393-01
Mercuric Nitrate Titration Cartridge, 2.256 N	each	921-01
Water, deionized	4 L	272-56

REQUIRED REAGENTS FOR THE SILVER NITRATE METHOD

Silver Nitrate Chloride Reagent Set (about 50 tests) Includes: (2) 1057-66, (1) 14396-01, (1) 14397-01		22880-00
Chloride 2 Indicator Powder Pillows	50/pkg	1057-66
Silver Nitrate Titration Cartridge, 0.2256 N	each	14396-01
Silver Nitrate Titration Cartridge, 1.128 N	each	14397-01
Water, deionized	4 L	272-56

REQUIRED APPARATUS FOR THE MERCURIC NITRATE METHOD AND SILVER NITRATE METHOD

Clippers, for opening pillows	each	968-00
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 250-mL	each	505-46
Select one or more based on sample concentration:		
Cylinder, graduated, 10-mL	each	508-38
Cylinder, graduated, 25-mL	each	508-40
Cylinder, graduated, 50-mL	each	508-41
Cylinder, graduated, 100-mL	each	508-42

OPTIONAL REAGENTS

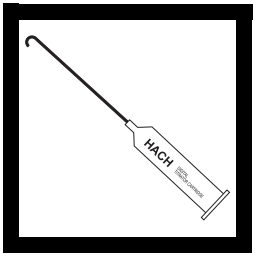
Chloride Standard Solution, 1000 mg/L Cl^-	500 mL	183-49
Chloride Standard Solution Voluette® Ampules, 12,500 mg/L Cl^-, 10-mL	16/pkg	14250-10
Hydrogen Peroxide, 30%, ACS	200 mL	144-45
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfide Inhibitor Powder Pillows	100/pkg	2418-99
Sulfuric Acid Standard Solution, 5.25 N	100 mL MDB	2449-32

OPTIONAL APPARATUS

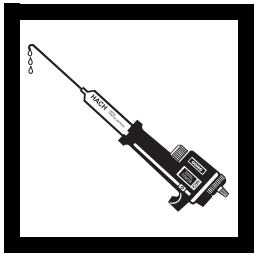
Description	Unit	Cat. No.
Bottle, wash, poly, 500-mL	each	620-11
Clamp, 2-prong extension, 38-mm	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473-mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	5/pkg	41578-00
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Funnel, poly, 65-mm	each	1083-67
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, 2-mL	each	14515-36
Pipet, volumetric, Class A, 5-mL	each	14515-37
Pipet, volumetric, Class A, 10-mL	each	14515-38
Pipet, volumetric, Class A, 20-mL	each	14515-20
Pipet, volumetric, Class A, 25-mL	each	14515-40
Pipet, volumetric, Class A, 50-mL	each	14515-41
Pipet, volumetric, Class A, 100-mL	each	14515-42
Pipet Filler, safety bulb	each	14651-00
*sens**ion**™1* Basic Portable pH Meter with electrode	each	51700-10
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10
Voluette® Ampule Breaker Kit	each	21968-00

CHLORINE, FREE AND TOTAL (0 to 3.00 mg/L as Cl_2)

DPD-FEAS Method



1. Insert a clean delivery tube into a 0.00564 N Ferrous Ethylenediammonium Sulfate (FEAS) Titration Cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step*, for assembly instructions, if necessary.



2. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

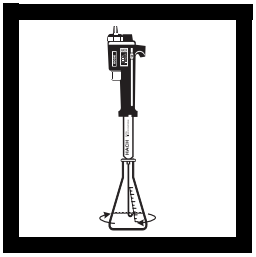
Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

3. Pipet 25.0 mL of sample into a 50-mL Erlenmeyer flask.

4. Add the contents of a DPD Free Chlorine Powder Pillow to the sample and swirl to mix.

Note: *Accuracy is unaffected if a small portion is undissolved.*

Note: *See Sampling and Storage following these steps.*

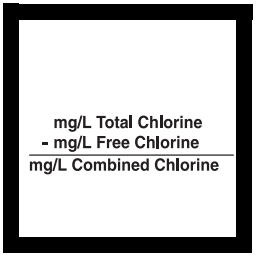


5. Place the delivery tube tip into the solution and swirl the flask while immediately titrating with FEAS to a colorless end point. Record the number of digits required.

Note: *Complete the titration rapidly.*

6. Calculate:

Digits Required x 0.01 = mg/L Free Chlorine



7. If total residual chlorine is desired, return to *step 3* and substitute a DPD Total Chlorine Powder Pillow in *step 4*. Wait three minutes before titrating. Continue with *step 5*. The results will be expressed as mg/L total chlorine.

mg/L Total Chlorine - mg/L Free Chlorine = mg/L Combined Chlorine

Sampling and Storage

Chlorine in water is easily lost. Therefore, start chlorine determinations immediately after sampling, avoiding excessive light and agitation. Do not store samples.

Accuracy Check

Standard Additions Method

This accuracy check should be performed when the analyst suspects interferences or to verify analytical technique.

1. Snap the neck off a Chlorine Standard Solution PourRite™ Ampule.
2. Use a TenSette® Pipet to add 0.10 mL, 0.20 and 0.30 mL of standard, respectively, to three 25-mL samples. Mix each well.
3. Analyze each sample as described in the procedure.
4. Each 0.1-mL addition of standard should require approximately 20 digits. Check the certificate enclosed with the PourRite Ampules to obtain the exact concentration. To determine the exact number of digits required for each 0.2-mL addition, multiply the exact concentration times the volume of the addition in mL times four. (Example: 50 mg/L x 0.1 mL x 4 = 20 digits.) If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

Interferences

Higher room temperatures tend to lead to higher free chlorine residual due to reaction of chloramines. Higher room temperatures also result in increased color fading. If the sample contains more than 250 mg/L alkalinity or 150 mg/L acidity as $CaCO_3$, the sample may not develop the full amount of color or it may instantly fade. To overcome this interference, adjust the pH of a separate 25-mL sample to a 6 to 7 pH by adding 1 N Sulfuric Acid Standard Solution or 1 N Sodium Hydroxide Standard Solution in small increments and using a pH meter. Record the amount of acid or base required. Add this amount of acid or base to the sample to be tested and proceed with *step 4*.

Bromine, iodine, ozone, and oxidized forms of manganese and chromium will also react and read as chlorine. To compensate for the effects of manganese, Mn^{4+}, or chromium, Cr^{6+}, add three drops of Potassium Iodide, 30 g/L to 25 mL of sample. Mix and wait one minute. Add three drops of Sodium Arsenite, 5 g/L and mix. Analyze this solution as described above. (If chromium is present, allow exactly the same reaction period in *step 7* with the DPD for both analyses.) Subtract the result from the original analysis to correct for the interference.

Summary of Method

The DPD-FEAS method provides a titrimetric procedure for determining free available chlorine and for estimating free and combined chlorine fractions present together. The magenta species, resulting from the oxidation of DPD by chlorine, is destroyed quantitatively by titration with ferrous ethylenediammonium sulfate and the volume of titrant required to reach a colorless end point is proportional to the chlorine concentration. Total residual chlorine may also be determined by this test.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Free and Total Chlorine Reagent Set (about 100 tests)		24453-00
Includes: (1) 14064-99, (1) 14070-99, (1) 22923-01		
DPD Free Chlorine Powder Pillows, 25 mL	100/pkg	14070-99
DPD Total Chlorine Powder Pillows, 25 mL	100/pkg	14064-99
Ferrous Ethylenediammonium Sulfate Titration Cartridge, 0.00564 N	each	22923-01

REQUIRED APPARATUS

Digital Titrator	each	16900-01
Flask, Erlenmeyer, 50-mL	each	505-41
Pipet, volumetric, Class A, 25-mL	each	14515-40
Pipet Filler, safety bulb	each	14651-00

OPTIONAL REAGENTS

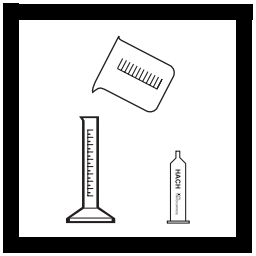
Chlorine Standard Solution, PourRite™ Ampules, 50-75 mg/L Cl_2, 2-mL	20/pkg	14268-20
Potassium Iodide Solution	100 mL MDB	343-32
Sulfuric Acid Standard Solution, 1.000 N	100 mL MDB	1270-32
Sodium Hydroxide Standard Solution, 1.000 N	100 mL MDB	1045-32
Sodium Arsenite Solution	100 mL MDB	1047-32

OPTIONAL APPARATUS

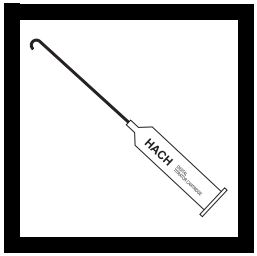
Clamp, 2-prong, extension	each	21145-00
Clamp Holder	each	326-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	5/pkg	41578-00
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette® Pipet	50/pkg	21856-96
PourRite™ Ampule Breaker	each	24846-00
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

CHLORINE, TOTAL

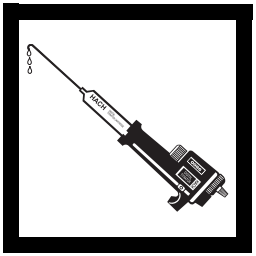
Iodometric Method (1 to 400 mg/L as Cl_2 Using Sodium Thiosulfate)



1. Select the sample volume and Sodium Thiosulfate Titration Cartridge corresponding to the expected chlorine concentration from *Table 1*.



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step*, for assembly instructions, if necessary.



3. Flush the delivery tube by turning the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Use a clean graduated cylinder to take a water sample. Pour sample into a clean 125- or 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water.

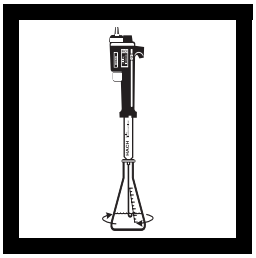
Note: *See Sampling and Storage following these steps.*

Table 1

Range (mg/L Cl_2)	Sample Volume (mL)	Titration Cartridge (N $Na_2S_2O_3$)	Catalog Number	Digit Multiplier
1-4	100	0.02256	24091-01	0.01
2-8	50	0.02256	24091-01	0.02
5-20	20	0.02256	24091-01	0.05
10-40	10	0.02256	24091-01	0.10
20-80	5	0.02256	24091-01	0.20
50-200	2	0.02256	24091-01	0.50
100-400	1	0.02256	24091-01	1.00

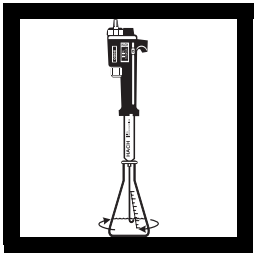
5. Add 2 Droppers (2 mL) Acetate Buffer Solution, pH 4 and swirl to mix.

6. Clip open the end of one Potassium Iodide Powder Pillow. Add the contents to the flask. Swirl to mix.

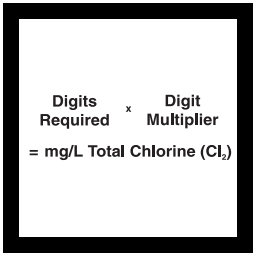


7. Place the delivery tube tip into the solution and swirl the flask while titrating with sodium thiosulfate until the solution is a pale yellow.

8. Add one dropper of starch indicator solution and swirl to mix. A dark blue color will develop.



9. Continue the titration until the solution changes from dark blue to colorless. Record the number of digits required.



10. Calculate:

Digits Required x Digit Multiplier = mg/L Total Chlorine (Cl2)

Note: *These procedures can be used to check iodine and bromine concentrations if chlorine is not present. Multiply the test result (in mg/L chlorine) by 3.58 or 2.25, respectively, to accurately express the iodine or bromine content of your sample.*

Sampling and Storage

Collect at least 200 mL of sample in a clean glass or polyethylene container. Analyze on site or as soon as possible after collection.

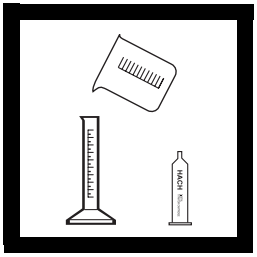
Accuracy Check

Standard Additions Method

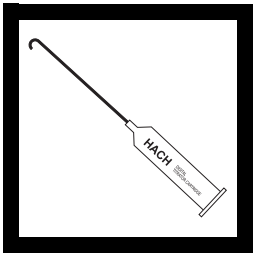
Perform this accuracy check when you suspect interferences or to verify analytical technique.

1. Snap the neck off a Chlorine Standard Solution PourRite™ Ampule.
2. Use a TenSette® Pipet to add 0.2 mL, 0.4 mL, and 0.6 mL of standard to three aliquots of sample of the same volume as used in the procedure.
3. Analyze each sample as described in the procedure.
4. Each 0.2-mL addition of standard should require approximately 10 digits of the titration cartridge solution. Check the certificate enclosed with the PourRite Ampules to obtain the exact concentration. To determine the exact number of digits required for each 0.2-mL addition, multiply the exact concentration times the volume of the addition in mL. (Example: 50 mg/L x 0.2 mL = 10 digits.) If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

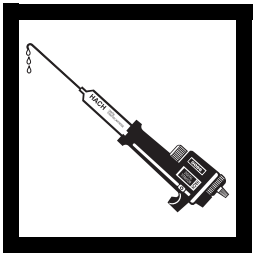
Iodometric Method (20 to 70,000 mg/L as Cl_2 Using Sodium Thiosulfate)



1. Select the sample volume and Sodium Thiosulfate Titration Cartridge corresponding to the expected chlorine concentration from *Table 2*.



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step*, for assembly instructions, if necessary.



3. Flush the delivery tube by turning the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TirtaStir® stirring apparatus. See General Description, Step 3 of Step-by-Step.*

4. Use a pipet or graduated cylinder to measure the sample volume from *Table 2*. Transfer the sample into a 125-mL Erlenmeyer flask and dilute to about the 50-mL mark with deionized water.

Table 2

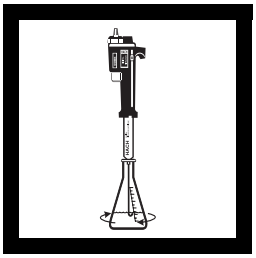
Range (mg/L Cl_2)	Sample Volume (mL)	Titration Cartridge (N $Na_2S_2O_3$)	Catalog Number	Digit Multiplier
20-80	25	0.113	22673-01	0.2
50-200	10	0.113	22673-01	0.5
100-400	5	0.113	22673-01	1
250-1000	2	0.113	22673-01	2.5
500-2000	1	0.113	22673-01	5
2000-9000 (0.2-0.9%)	4	2.00	14401-01	22.2
5000-18,000 (0.5-1.8%)	2	2.00	14401-01	44.3
10,000-35,000 (1.0-3.5%)	1	2.00	14401-01	88.7
20,000-70,000 (2.0-7.0%)	0.5	2.00	14401-01	177

5. Add the contents of one Dissolved Oxygen 3 Powder Pillow.

Note: *Normally the addition of the powder pillow will lower the pH to 4 or less. If the sample size is large and highly alkaline, verify the solution pH is 4 or less with a pH meter or pH paper before proceeding.*

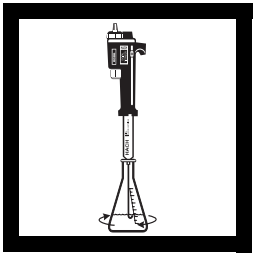
6. If you are using the 2.00 N titration cartridge, add the contents of one Potassium Iodide Powder Pillow (Cat. No. 20599-96) to the flask and swirl to mix.

If you are using the 0.113 N titration cartridge, add the contents of one Potassium Iodide Powder Pillow (Cat. No. 1077-99) to the flask and swirl to mix.

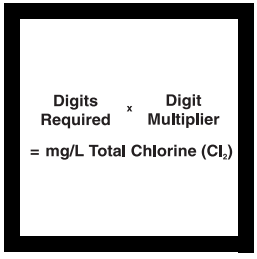


7. Place the delivery tube tip into the solution and swirl the flask while titrating with sodium thiosulfate until the solution is a pale yellow.

8. Add one dropperful of starch indicator solution and swirl to mix. A dark blue color will develop.



9. Continue the titration until the solution changes from dark blue to colorless. Record the number of digits required.



10. Calculate:

Digits Required x Digits Multiplier = mg/L Total Chlorine (Cl2)

To convert the above results to the equivalent percent chlorine (Cl_2), divide by 10,000.

Accuracy Check

Standard Additions Method

This accuracy check is applicable **only for the 0.113 N titration cartridge**. Perform it when interferences are suspected or to verify analytical technique.

1. Snap the neck off a Chlorine Standard Solution PourRite Ampule.
2. Use a TenSette Pipet (or glass pipet) to add 1.0 mL, 2.0 mL, and 3.0 mL of standard to three samples of the same volume as used in the procedure.
3. Analyze each sample as described in the procedure.
4. Each 1.0-mL addition of standard should require approximately 10 digits of the 0.113 N titration cartridge. Check the certificate enclosed with the PourRite Ampules to obtain the exact concentration. To determine the exact number of digits required for each 1.0-mL addition, multiply the exact concentration times the volume of the addition in mL. Divide this by five. For example: (50 mg/L x 1.0 mL) ÷ 5 = 10 digits. If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

Summary of Method

Total chlorine concentration equals the concentration of the free and the combined forms of chlorine. Free chlorine reacts readily with ammonia to form combined chlorine such as monochloramines. When potassium iodide is added to a sample containing chlorine at a pH less than 8, free iodine is liberated in direct proportion to the amount of total chlorine present. The iodine is then titrated with sodium thiosulfate.

REQUIRED REAGENTS (For Using the 0.02256 N Titration Cartridge)

Description	Unit	Cat. No.
Acetate Buffer Solution, pH 4	100 mL MDB	14909-32
Potassium Iodide Powder Pillows	100/pkg	1077-99
Starch Indicator Solution	100 mL MDB*	349-32
Sodium Thiosulfate Titration Cartridge, 0.02256 N	each	24091-01

REQUIRED REAGENTS (For Using the 0.113 N Titration Cartridge)

Chlorine Reagent Set, 20-2,000 mg/L (about 100 tests) Includes: (1) 349-32, (1) 987-99, (1) 1077-99, (1) 22673-01		22725-00
Dissolved Oxygen 3 Powder Pillows	100/pkg	987-99
Potassium Iodide Powder Pillows	100/pkg	1077-99
Sodium Thiosulfate Titration Cartridge, 0.113 N	each	22673-01
Starch Indicator Solution	100 mL MDB*	349-32
Water, deionized	4 L	272-56

REQUIRED REAGENTS (For Using the 2.00 N Titration Cartridge)

Chlorine Reagent Set, 2,000-70,000 mg/L (about 100 tests) Includes: (1) 349-32, (1) 987-99, (2) 14401-01, (2) 20599-96		24448-00
Dissolved Oxygen 3 Powder Pillows	100/pkg	987-99
Potassium Iodide Powder Pillows	50/pkg	20599-96
Sodium Thiosulfate Titration Cartridge, 2.00 N	each	14401-01
Starch Indicator Solution	100 mL MDB*	349-32
Water, deionized	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening pillows	each	968-00
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 125 mL	each	505-43
Pipet Filler, 3-valve	each	12189-00
Select one or more based on sample concentration:		
Pipet, serological, 1 mL	each	532-35
Pipet, volumetric, Class A, 1 mL	each	14515-35
Pipet, volumetric, Class A, 2 mL	each	14515-36
Pipet, volumetric, Class A, 4 mL	each	14515-04
Pipet, volumetric, Class A, 5	each	14515-37
Pipet, volumetric, Class A, 10 mL	each	14515-38
Pipet, volumetric, Class A, 25 mL	each	14515-40

* Contact Hach for larger sizes.

OPTIONAL REAGENTS

Description	Unit	Cat. No.
Chlorine Standard Solution, PourRite™ Ampules, 50-75 mg/L as Cl_2, 2 mL	20/pkg	14268-20

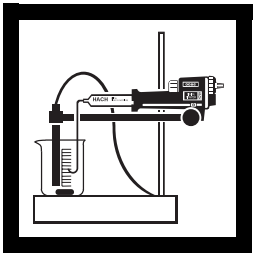
OPTIONAL APPARATUS

Clamp, 2-prong, extension, 38 mm	each	21145-00
Clamp Holder	each	326-00
Cylinder, graduated, 5 mL	each	508-37
Cylinder, graduated, 10 mL	each	508-38
Cylinder, graduated, 25 mL	each	508-40
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	5/pkg	41578-00
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette® Pipet	50/pkg	21856-96
pH Paper, 1-11 pH	5 rolls/pkg	391-33
PourRite™ Ampule Breaker	each	24846-00
*sens**ion**™1* Basic Portable pH Meter with electrode	each	51700-10
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

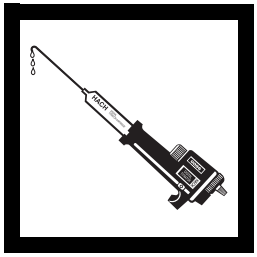
CHLORINE, FREE (0 to 1000 µg/L as Cl_2)

Amperometric Forward Titration

USEPA Accepted for Reporting*



1. Assemble the Amperometric Digital Titrator System according to the instructions in the *Amperometric Titrator Instruction Manual*.



2. Install the 0.00564 N Phenylarsine Oxide (PAO) cartridge. Flush the Digital Titrator delivery tube by turning the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *When a new probe is placed in service or when the probe has not been used recently, prepare it according to the Probe Stabilization instructions in the Amperometric Titrator Instruction Manual.*

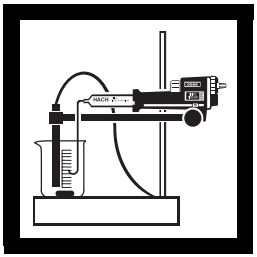
3. With minimum agitation, measure 200 mL of sample with a clean graduated cylinder. Transfer the sample to a clean 250-mL beaker containing the 50-mm stirring bar supplied with the system.

Note: *An improper stirring bar size can result in volatilization of chlorine, instability of readings and loss of sensitivity.*

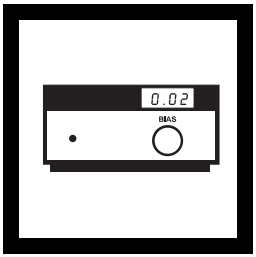
4. Add 1 mL of pH 7 Phosphate Buffer Solution.

Note: *If the sample pH is between 6.5 and 7.5 it is not necessary to add the buffer.*

* Procedure is equivalent to *Standard Methods for the Examination of Water and Wastewater* (18th ed.) 4500 Cl D for drinking water.

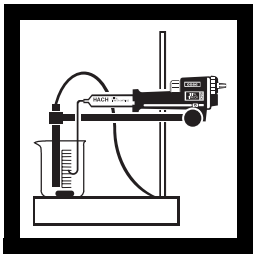


5. Place the beaker on the TitraStir® Stir Plate and immerse the tips of the probe and delivery tube in the solution. The probe's platinum wires must be submerged. Turn on the stirring motor.



6. Note the LED reading on the Amperometric Titrator. Unlock the BIAS control and adjust the BIAS control knob until a reading between 0.50-0.60 is obtained. Lock the BIAS control.

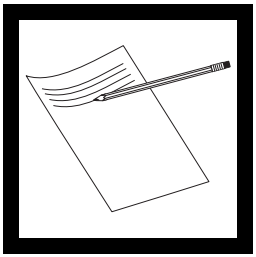
Note: *The bias adjustment controls the slope of the titration curve. The actual instrument reading is not important; but rather the change in the readings as the titration proceeds. The adjustment need not be precise.*



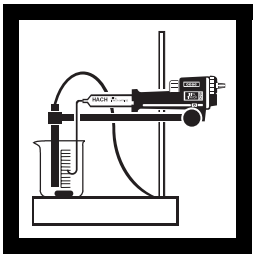
7. Using the Digital Titrator delivery knob, dispense the PAO titrant Solution in 5-10 digit increments while noting the LED reading.

Note: *If the chlorine content of the sample is high, add titrant at a faster rate; only the end point of the titration and the volume of titrant used at the end point are of concern. For example, if the chlorine content is approximately 500 µg/L, up to 300 digits of 0.00564 N PAO could be added at once. As the end point is approached, dispense in small increments.*

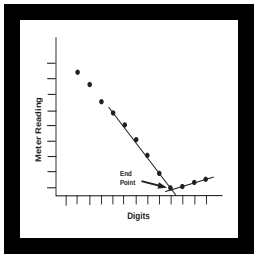
Note: *If excess reductant such as sulfite, bisulfite or sulfur dioxide is present in the sample, the LED readings will not decrease and may even increase. This indicates that no free chlorine is present in the sample*



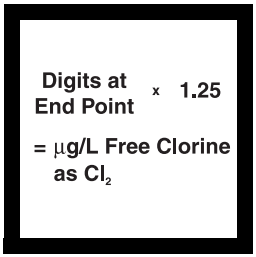
8. As the end point of the titration is approached, record the LED readings along with the corresponding digits displayed on the Digital Titrator counter. Near the titration end point, add 2 to 5 digits of titrant; wait a few seconds for a stable reading and record.



9. Continue the titration, recording at least three points on the downward sloping curve and at least three points after the end point has been reached. The latter points will have little change in the LED readings.



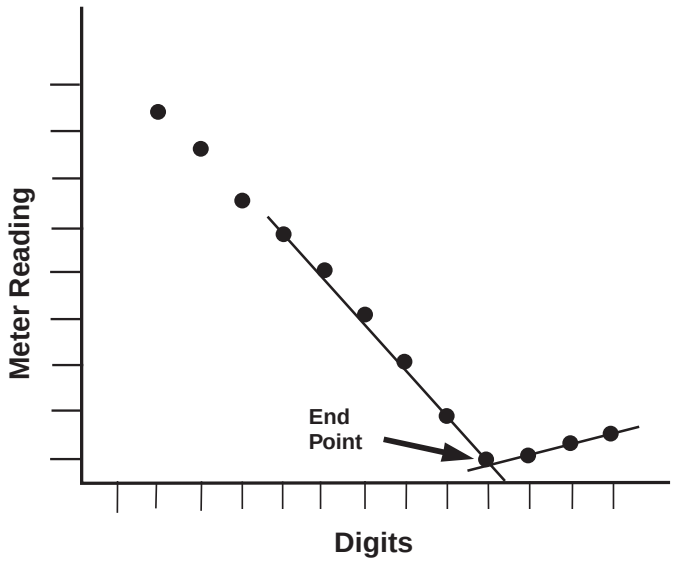
10. Using linear graph paper, plot the recorded readings from the Amperometric Titrator on the vertical axis and the corresponding Digital Titrator digits on the horizontal axis. Draw the two best intersecting lines through the points; see *Figure 1*. Determine the number of digits at the intersection of the lines; this is the end point.



11. Calculate the µg/L free chlorine:

Digits at End Point x 1.25 = µg/L free chlorine as Cl_2

Figure 1
Sample Plot



Accuracy Check

Standard Additions Method*

1. Snap the top off a Chlorine Standard Solution PourRite™ Ampule. Note the certificate value of the standard in mg/L.
2. Split a fresh sample into two 200-mL portions.
3. Using a TenSette® Pipet, add from 0.1 to 0.5 mL of the standard to one portion and swirl to mix. This is the *spiked sample*.
4. Analyze both the sample and spiked sample and record the chlorine concentration of each.
5. Calculate the theoretical concentration of the spiked sample:

$$\text{heoretical concentration} = \frac{(C_u \times V_u) + (C_s \times V_s)}{V_u + V_s}$$

Where:

C_u = measured concentration of sample, in mg/L (µg/L divided by 1000)

V_u = volume of sample in mL

C_s = concentration of chlorine standard (mg/L, certificate value)

V_s = volume of standard added in mL

6. Calculate the percent spiked recovery:

$$\text{Spike Recovery} = \frac{\text{Spiked sample result, in mg/L}}{\text{Theoretical concentration calculated, in mg/L}} \times 100$$

Example:

Sample result (C_u) = 120 µg/L or 0.120 mg/L

Spiked sample result = 185 µg/L or 0.185 mg/L

Volume Sample (V_u) = 200 mL

Volume Standard (V_s) = 0.2 mL

Chlorine Standard (C_s) = 68.1 mg/L

$$\text{heoretical concentration} = \frac{(0.120 \times 200) + (68.1 \times 0.2)}{200 + 0.2} = 0.188 \text{ mg/L}$$

$$\text{\% Spike recovery} = \frac{0.185 \text{ mg/L}}{0.188 \text{ mg/L}} \times 100 = 98\%$$

Ideally, the percent recovery should be 100%. Generally, results from 80-120% recovery are considered acceptable.

* The standard additions technique is not applicable for samples containing excess reducing agents such as sulfur dioxide, sulfite, or bisulfite.

Precision

In a single laboratory, using a standard solution of 338 µg/L chlorine, a single operator obtained a standard deviation of ± 5.2 µg/L chlorine.

Detection Limit

With good operator technique, the estimated detectable concentration is approximately 15 µg/L chlorine using 0.00564 N PAO.

Sampling and Storage

Chlorine is rapidly lost from water. Avoid exposure to sunlight or other strong light. Avoid excessive agitation. Analyze samples immediately.

Interferences

- Silver ions poison the electrode.
- Copper ions interfere.
- Interferences are sometimes found in highly turbid water and those containing surface active agents.
- Oxidized manganese and other oxidizing reagents give positive interferences.
- Some uncertainty in the end point may be observed with samples containing high organic content.
- Samples containing excess reducing agents, such as sulfur dioxide, sulfite, and bisulfite do not contain free chlorine and can not be titrated under the conditions of the test.
- Highly buffered samples or extreme sample pH may exceed the buffering capacity of the buffer reagent. If necessary, add additional buffer and check pH of sample prior to titration.

Summary of Method

In the amperometric forward titration procedure for free chlorine, a small electrical current is applied across two identical platinum electrodes. No current can flow between the electrodes unless a

substance that can be oxidized at the anode and a substance that can be reduced at the cathode are both present. In the case of the free chlorine titration with phenylarsine oxide (PAO), chlorine is reduced at the cathode to chloride due to the addition of PAO and PAO is oxidized from the +3 oxidation state to the +5 oxidation state at the anode. Prior to the end point of the titration, both free chlorine and chloride are present in solution; allowing current to flow, even with a very small applied potential. At the end point, no free chlorine remains and the solution cannot conduct even if excess PAO titrant is added. The end point is defined when no change in current occurs, signaling all free chlorine has been reacted.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Phenylarsine Oxide Solution, 0.00564 N Digital Titrator Cartridge	each	1999-01
Phosphate Buffer Solution, pH 7	100 mL MDB	21553-32

REQUIRED APPARATUS

Amperometric Titrator Assembly	each	19299-00
Digital Titrator	each	16900-01
Beaker, low-form, 250 mL	each	500-46
Cylinder, graduated, 250 mL	each	508-46
Delivery Tubes, 90° with hook	5/pkg	41578-00
Probe Assembly, Amperometric Titrator	each	19390-00
Stir Bar, octagonal, Teflon-coated, 50.8 x 7.9 mm	each	20953-55
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

OPTIONAL REAGENTS

Chlorine Standard Solution PourRite™ Ampules, 50-75 mg/L Cl_2, 2 mL	20/pkg	14268-20
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

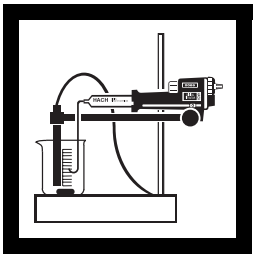
Pipet, TenSette® 0.1 to 1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette® Pipet	50/pkg	21856-96
PourRite™ Ampule Breaker	each	24846-00
Standard Methods for the Examination of Water and Wastewater, 19th edition	each	22708-00

CHLORINE, TOTAL (6 to 1000 µg/L as Cl_2)

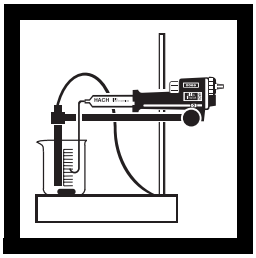
Amperometric Back Titration

USEPA Accepted for Reporting*

Phase 1: Adjusting the Electrode Response Slope



1. Assemble the Amperometric Digital Titrator System according to the instructions in the *Amperometric Titrator Instruction Manual*.



2. Install the Standard Iodine Titrant Cartridge, 0.028 N. Flush the Digital Titrator delivery tube by turning the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

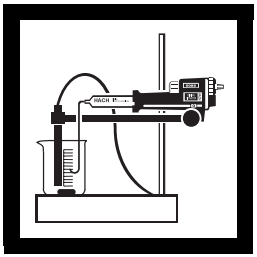
Note: *When a new probe is used or the probe has not been used recently, prepare it according to the Probe Stabilization instructions in the Amperometric Titrator Instruction Manual.*

3. Using a graduated cylinder, measure 200 mL of deionized water into a clean 250-mL beaker. Place the 50-mm stirring bar supplied with the system into the beaker.

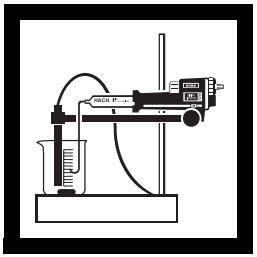
Note: *An improper size stirring bar can result in volatilization of iodine, instability of readings and loss of sensitivity.*

4. Add 1 mL of pH 4 Acetate Buffer and the contents of one Potassium Iodide Pillow.

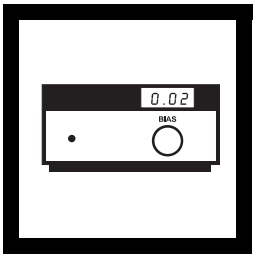
* Procedure is equivalent to USEPA method 330.2 and *Standard Methods for the Examination of Water and Wastewater* (17th ed.) 4500-Cl C for wastewater.



5. Place the beaker on the TitraStir® Stir Plate and immerse the tips of the probe and delivery tube in the solution. The probe's platinum wires must be submerged. Turn on the stirring motor.



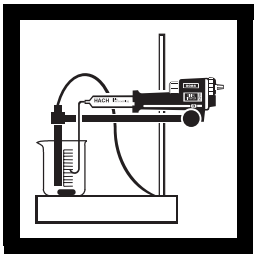
6. Using the Digital Titrator delivery knob, add 50 digits of Standard Iodine Titrant Solution.



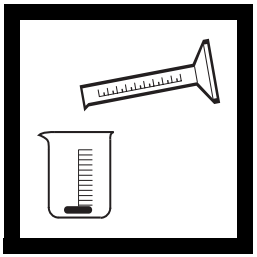
7. Note the LED reading on the Amperometric Titrator. Unlock the BIAS control and adjust the BIAS control knob until a stable reading between 0.50-0.60 is obtained. Lock the BIAS control.

8. Remove the probe arm from the beaker and rinse the platinum wires with deionized water. Adjustment of the electrode response slope is complete.

Phase 2: Standardization of the Iodine Titrant

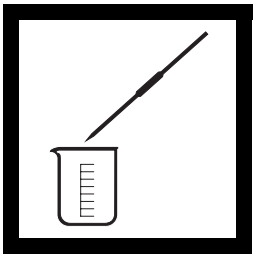


1. Set-up the Amperometric Digital Titrator System as in *Phase 1: Adjusting the Electrode Response Slope* if it has not already been done. Reset the Digital Titrator counter to zero and wipe the tip.



2. Using a graduated cylinder, measure 200 mL of deionized water into a clean 250-mL beaker. Place the 50-mm stirring bar supplied with the system into the beaker.

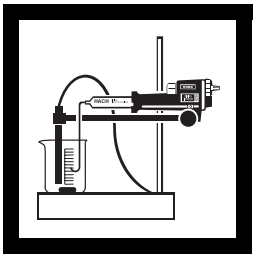
Note: *An improper size of stirring bar can result in volatilization of iodine, instability of readings and loss of sensitivity.*



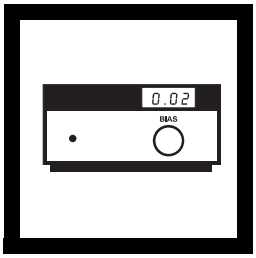
3. Using a Class A pipet, transfer 1.00 mL of 0.00564 N Sodium Thiosulfate Solution to the beaker. Swirl to mix.

Note: *Alternatively, use 0.00564 N Phenylarsine Oxide (PAO), Cat. No. 1999, instead of thiosulfate.*

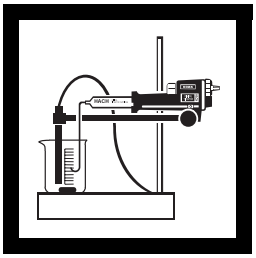
4. Add 1 mL of pH 4 Acetate Buffer Solution and the contents of one Potassium Iodide Powder Pillow.



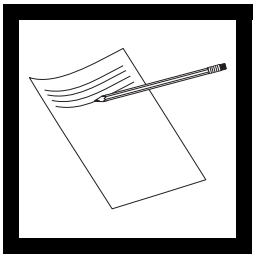
5. Place the beaker on the TitraStir Stir Plate and immerse the tips of the probe and delivery tube in the solution. The probe's platinum wires must be submerged. Turn on the stirring motor.



6. Note the LED reading on the Amperometric Titrator. It should read 0.00 ± 0.05. DO NOT adjust the BIAS control.

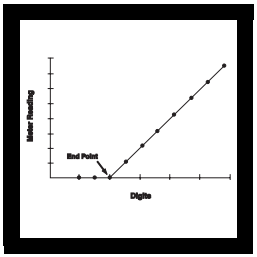


7. Using the Digital Titrator delivery knob, dispense 100 digits of Standard Iodine Titrant Solution and note the LED reading.

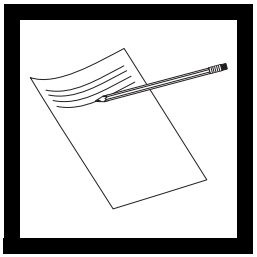


8. Continue dispensing titrant in 5-10 digit increments while noting the LED reading. Record at least 3 points (null current values and Digital Titrator reading), before the end point is reached. After the end point of the titration (nominal 160 digits), record the increasing LED readings along with the corresponding digits displayed on the Digital Titrator counter. Add 5-10 digits of titrant; wait a few seconds for a stable reading and record it. Stop adding titrant when the LED readings exceed 0.60.

Note: *LED readings above 0.60 will be excessively noisy.*



9. Using linear graph paper, plot the recorded readings from the Amperometric Titrator on the vertical axis and the corresponding Digital Titrator digits on the horizontal axis. Draw the two best intersecting lines through the points plotted. See *Figure 1*. Determine the number of digits at the intersection of the lines. This is the *standard* end point.



10. Record the standard end point digits value. This value will be used in calculation of the sample chlorine concentration.

Note: *The iodine titrant concentration is approximately 0.0282 N, which relates to 160 digits needed to titrate 1.00 mL of 0.00564 N Thiosulfate. If the calculated end point is greater than 160 digits, this indicates the Standard Iodine Titrant is weaker than when packaged. Discard the Standard Iodine Titrant cartridge if the calculated standardization end point is greater than 200 digits.*

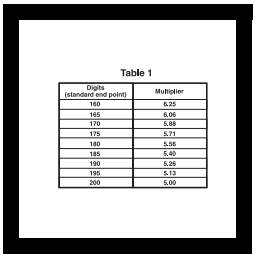
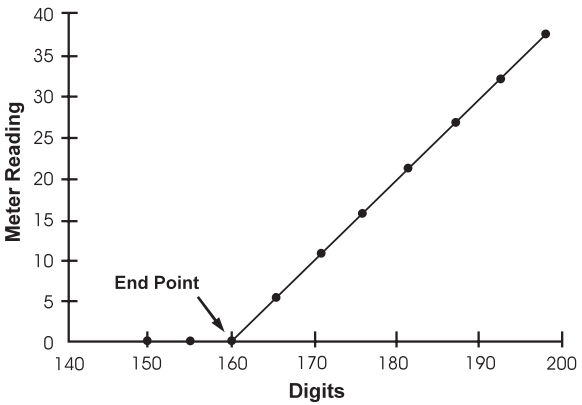


Table 1

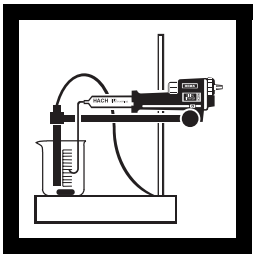
Digits (standard end point)	Multiplier
160	6.25
165	6.06
170	5.88
175	5.71
180	5.56
185	5.40
190	5.26
195	5.13
200	5.00

11. Locate the appropriate multiplier based on the standard end point in *Table 1* on page 93. The multiplier is used in *Phase 3: Titration of Sample for Total Residual Chlorine*. Interpolation between values in the table is not necessary.

igure 1
ack Amperometric
itration Graph



Phase 3: Titration of Sample for Total Residual Chlorine



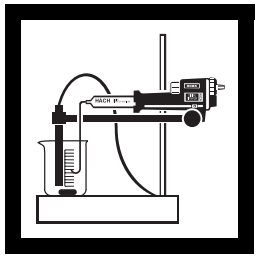
1. Set-up the Amperometric Digital Titrator System as in *Phase 1: Adjusting the Electrode Response Slope* if it has not already been done. Reset the Digital Titrator counter to zero and wipe the tip.

2. Place a clean 50-mm stirring bar supplied with the system into a clean 250-mL beaker. Using a Class A pipet, transfer 1.00 mL of 0.00564 N Sodium Thiosulfate Solution to the a beaker. Add 1 mL of pH 4 Acetate Buffer Solution to the beaker.

Note: *An improper size stirring bar can result in volatilization of chlorine, instability of readings and loss of sensitivity. Alternatively, use 0.00564 N Phenylarsine Oxide (PAO), Cat. No. 1999, instead of thiosulfate.*

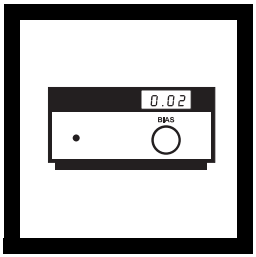
3. With minimum agitation, measure 200 mL sample with a clean graduated cylinder and transfer the sample to the beaker. Swirl to mix the reagents with sample.

Note: *Steps 2-3 can be performed at the sampling site thereby "fixing" the sample for later analysis. Pipet 1.00 mL of 0.00564 N Sodium Thiosulfate and add 1.0 mL of Acetate Buffer into a clean, dry glass sampling bottle (e.g. BOD bottle). At the sample site, measure 200 mL of sample with a graduated cylinder and transfer to the sampling bottle. Swirl to mix. Before analysis, quantitatively transfer the entire contents of the sampling bottle to the 250-mL beaker. Minimize delay between sampling and analysis (1 hour maximum) to prevent decomposition of thiosulfate in the sample.*

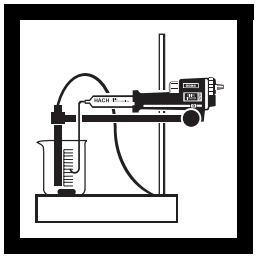


4. Place the beaker on the TitraStir Stir Plate and immerse the tips of the probe and delivery tube in the solution. The probe's platinum wires must be submerged. Turn on the stirring motor.

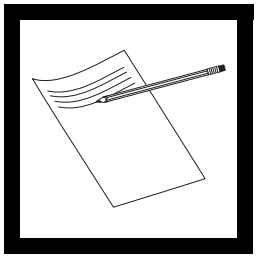
5. Add the contents of one pillow of Potassium Iodide Reagent to the beaker and allow the powder to dissolve.



6. Note the LED reading on the Amperometric Titrator. It should read 0.00 ± 0.05. DO NOT adjust the BIAS control.

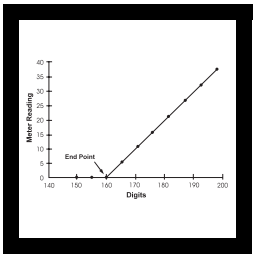


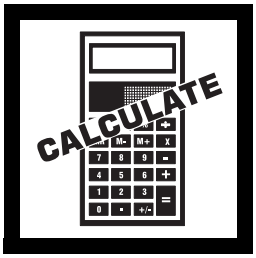
7. Using the Digital Titrator delivery knob, dispense the Standard Iodine Titrant Solution in 5-10 digit increments while noting the LED reading. Record at least 3 points (null current values and Digital Titrator reading), before end point is reached.



8. After the end point of the titration is reached, record the increasing LED readings along with the corresponding digits displayed on the Digital Titrator counter. Add 5-10 digits of titrant; wait a few seconds for a stable reading and record. Stop the titrant addition when the LED readings exceed 0.60.

Note: *LED readings above 0.60 will be excessively noisy. With samples containing excess de-chlorinating agents, such as sulfur dioxide, sulfite or bisulfite, the titration end point (number of digits) will be greater than the number of digits obtained during the standardization. It is not necessary to continue the titrant addition if the number of digits used in the sample titration exceeds that calculated for the standardization end point. This indicates that no free or combined chlorine is present in the sample.*





9. Using linear graph paper, plot the recorded readings from the Amperometric Titrator on the vertical axis and the corresponding Digital Titrator digits on the horizontal axis. Draw the two best intersecting lines through the points plotted. See *Figure 1* on page 90. Determine the number of digits at the intersection of the lines. This is the *sample* end point.

10. Calculate the µg/L total chlorine:

[Digits (Standard End Point) - Digits (Sample End Point)] x Multiplier = µg/L Cl_2
(Multiplier is from Phase 2.)

Example: Standard EP = 160 digits
Multiplier = 6.25
Sample EP = 150 digits

µg/L total chlorine =
[160 - 150] x 6.25 =
10 x 6.25 = 63 (round up)

Note: *To preserve the strength of the iodine titrant solution, always remove the delivery tube from the Digital Titrator cartridge and replace the cap when not in use. Protect the iodine titrant solution from direct sunlight.*

Table 1

Digits (standard end point)	Multiplier
160	6.25
165	6.06
170	5.88
175	5.71
180	5.56
185	5.40
190	5.26
195	5.13
200	5.00

Sampling and Storage

Chlorine is rapidly lost from water. Avoid exposure to sunlight or other strong light. Avoid excessive agitation. Analyze samples immediately or fix the sample by pre-addition of standard thiosulfate and buffer as indicated in *Phase 3: Titration of Sample for Total Residual Chlorine*. The fixing procedure should be used for brief transportation delays—not for storage of samples.

Accuracy Check

Standard Additions Method*

Snap the top off a Chlorine Standard Solution PourRite™ Ampule. Note the certificate value of the standard in mg/L.

1. Split a fresh sample into two 200-mL portions.

2. Using a TenSette® Pipet, add from 0.1 to 0.5 mL of the standard to one portion and swirl to mix. This is the *spiked sample*.

3. Analyze each sample as described above and record the chlorine concentrations.

4. Calculate the theoretical concentration of the spiked sample:

$$\text{heoretical concentration} = \frac{(C_u \times V_u) + (C_s \times V_s)}{V_u + V_s}$$

Where:

C_u = measured concentration of sample, in mg/L (µg/L divided by 1000)

V_u = volume of sample in mL

C_s = concentration of chlorine standard (mg/L, certificate value)

V_s = volume of standard added in mL

5. Calculate the percent spiked recovery:

$$\% \text{ Spike recovery} = \frac{\text{Spiked sample result, in mg/L}}{\text{Theoretical concentration calculated, in mg/L}} \times 100$$

* Standard additions is not applicable for samples containing excess reducing agents such as sulfur dioxide, sulfite, or bisulfite.

Example:

Sample result (C_u) = 120 µg/L or 0.120 mg/L

Spiked sample result = 185 µg/L or 0.185 mg/L

Volume Sample (V_u) = 200 mL

Volume Standard (V_s) = 0.2 mL

Chlorine Standard (C_s) = 68.1 mg/L

$$\text{heoretical concentration} = \frac{(0.120 \times 200) + (68.1 \times 0.2)}{200 + 0.2} = 0.188 \text{ mg/L}$$

Ideally, the percent recovery should be 100%. Generally, results from 80-120% recovery are considered acceptable.

Precision

In a single laboratory, using a standard solution of 120 µg/L chlorine, a single operator obtained a standard deviation of ± 19 µg/L chlorine.

Detection Limit

The estimated detectable concentration is equivalent to one digit of 0.0282 N Standard Iodine Titrant Solution or approximately 6 µg/L chlorine.

Interferences

- Silver ions poison the electrode.
- Copper ions interfere.
- Interferences are sometimes found in highly turbid water and those containing surface active agents.
- Oxidized manganese and other oxidizing reagents give positive interferences.
- Some uncertainty in the end point may be observed with samples containing high organic content.
- Iron and nitrite interference are minimized by buffering to pH 4 before adding potassium iodide.

- In samples containing excess reducing agents, such as sulfur dioxide, sulfite, and bisulfite, the titration end point will be shifted, indicating the sample contains no free or combined chlorine.

- Highly buffered samples or extreme sample pH may exceed the buffering capacity of the buffer reagent. If necessary, add additional buffer and check the pH of the sample prior to titration.

Summary of Method

The back titration procedure minimizes errors caused by liberating the full concentration of iodine in the sample and is the preferred method for amperometric measurement for total chlorine in wastewaters. In the back titration procedure, the end point signal is reversed because the remaining thiosulfate (or phenylarsine oxide) added to the sample is titrated with standard iodine. The end point of the back titration is reached just when free iodine exists in the sample resulting in a measurable polarization current. The end point is estimated by continued addition of titrant, recording of the current at each titrant addition, and graphing the data points. Where the best line between the data points intersects with the null current, the number of digits (from the Digital Titrator) at the end point can be determined and the concentration of chlorine calculated.

It is necessary to adjust the electrode sensitivity by using the bias control prior to performing the analysis. The bias adjustment is set by adding a known amount of standard iodine titrant to deionized water and adjusting the bias control to a given value on the display. The electrode sensitivity will vary depending on the probe conditioning. Adjustment should be made at least daily or before each series of samples.

Although the iodine titrant solution is formulated and packaged to be quite stable it is recommended the iodine be routinely standardized against standard thiosulfate or phenylarsine oxide. The number of digits determined for the iodine standardization is recorded and used in the calculation of the sample's chlorine concentration.

To preserve the strength of the iodine titrant solution, always remove the delivery tube from the Digital Titrator cartridge and replace the cap when not in use. Protect the iodine titrant solution from direct sunlight.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Acetate Buffer Solution, pH 4.0	100 mL MDB	14909-32
Potassium Iodide Powder Pillows	100/pkg	1077-99
Standard Iodine Titrant Solution, 0.028 N	each	23333-01
Sodium Thiosulfate Standard Solution, 0.00564 N	100 mL	24088-42

REQUIRED APPARATUS

Amperometric Titrator Assembly	each	19299-00
Beaker, low-form, 250-mL	each	500-46
Cylinder, graduated, 250-mL	each	508-46
Digital Titrator	each	16900-01
Delivery Tubes, 90° with hook	5/pkg	41578-00
Pipet, volumetric, Class A, 1-mL	each	14515-35
Probe Assembly, Amperometric Titrator	each	19390-00
Stir Bar, octagonal, Teflon-coated, 50.8 x 7.9 mm	each	20953-55
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

OPTIONAL REAGENTS

Chlorine Standard Solution PourRite™ Ampules, 50-75 mg/L Cl_2, 2-mL	20/pkg	14268-20
Phenylarsine Oxide Solution, 0.00564 N	100 mL	1999-42
Water, deionized	4 L	272-56

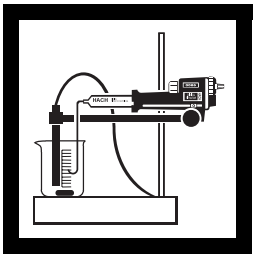
OPTIONAL APPARATUS

Bottle, BOD, 300-mL	each	621-00
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette® Pipet	50/pkg	21856-96
PourRite™ Ampule Breaker	each	24846-00

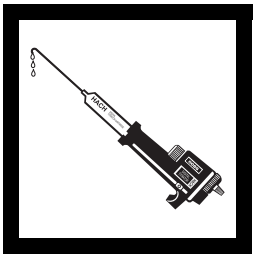
CHLORINE, TOTAL (0 to 1000 µg/L as Cl_2) For drinking water or wastewater

Amperometric Forward Titration

USEPA Accepted for Reporting*



1. Assemble the Amperometric Digital Titrator System according to the instructions in the *Amperometric Titrator Instruction Manual*.



2. Install the Phenylarsine Oxide (PAO), 0.00564 N Cartridge. Flush the Digital Titrator delivery tube by turning the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *When a new probe is used or when the probe has not been used recently, prepare it according to the Probe Stabilization instructions in the Amperometric Titrator Instruction Manual.*

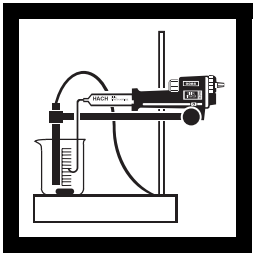
3. With minimum agitation, measure 200 mL sample with a clean graduated cylinder. Transfer the sample to a clean 250-mL beaker containing the 50-mm stirring bar supplied with the system.

Note: *An improper size of stirring bar can result in volatilization of chlorine, instability of readings and loss of sensitivity.*

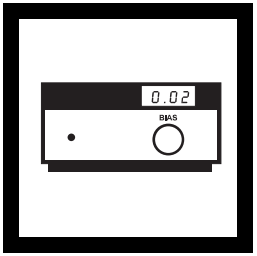
4. Add the contents of one Potassium Iodide Powder Pillow and swirl to dissolve.

* Procedure is equivalent to USEPA method 330.1 and 330.3, *Standard Methods for the Examination of Water and Wastewater* (18th ed.) 4500-Cl D for drinking water and *Standard Methods* (17th ed.) 4500-Cl D for wastewater.

5. Add 1 mL of pH 4 Acetate Buffer Solution.

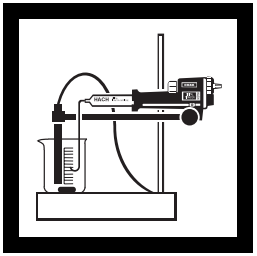


6. Place the beaker on the TitraStir® Stir Plate and immerse the tips of the probe and delivery tube in the solution. The probe's platinum wires must be submerged. Turn on the stirring motor.



7. Note the LED reading on the Amperometric Titrator. Unlock the BIAS control and adjust the BIAS control knob until a reading between 0.50-0.60 is obtained. Lock the BIAS control.

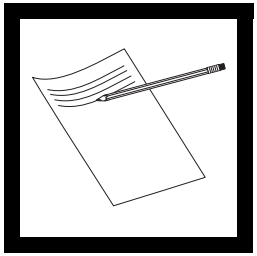
Note: *The bias adjustment controls the slope of the titration curve. The actual instrument reading is not important; but rather the change in the readings as the titration proceeds. The adjustment need not be precise.*



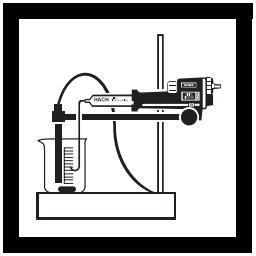
8. Using the Digital Titrator delivery knob, dispense the PAO titrant Solution in 5-10 digit increments while noting the downward reading.

Note: *If the chlorine content of the sample is high, add titrant at a faster rate; only the end point of the titration and the volume of titrant used at the end point are of concern. For example, if the chlorine content is approximately 500 µg/L, up to 300 digits of 0.00564 N PAO could be added at once. As the end point is approached, dispense in small increments.*

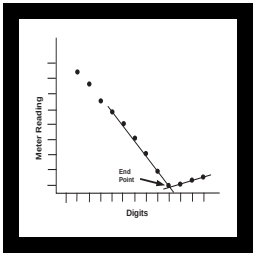
Note: *If excess reductant, such as sulfite, bisulfite, or sulfur dioxide is present in the sample, the LED readings will not decrease and may even increase. This indicates that no free chlorine or chloramines are present in the sample.*



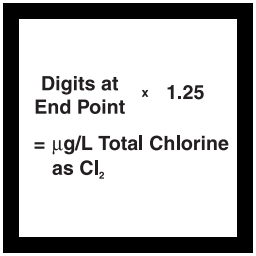
9. As the end point of the titration is approached, record the LED readings along with the corresponding digits displayed on the Digital Titrator counter. Near the titration end point, add 2 to 5 digits of titrant; wait a few seconds for a stable reading and record.



10. Continue the titration, recording at least three points on the downward sloping curve and at least three points after the end point has been reached. The latter points will have little change in the LED readings.



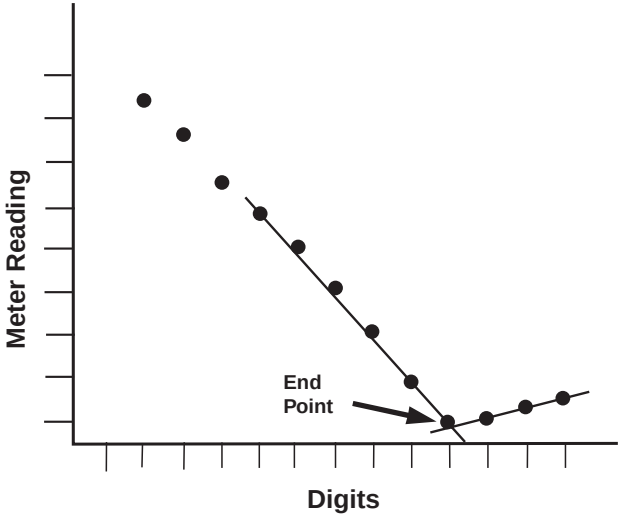
11. Using linear graph paper, plot the recorded readings from the Amperometric Titrator on the vertical axis and the corresponding Digital Titrator digits on the the on the horizontal axis. Draw the two best intersecting lines through the points see *Figure 1*. Determine the number of digits at the intersection of the lines; this is the end point.



12. Calculate the µg/L total chlorine:

Digits at End Point X 1.25 = µg/L total chlorine as Cl_2

Figure 1
Sample Plot



Accuracy Check

Standard Additions Method*

1. Snap the top off a Chlorine Standard Solution PourRite™ Ampule. Note the certificate value of the standard in mg/L.
2. Split a fresh sample into two 200-mL portions.
3. Using a TenSette® Pipet, add from 0.1 to 0.5 mL of the standard to one portion and swirl to mix. This is the *spiked sample*.
4. Analyze both the sample and spiked sample and record the concentration of each.
5. Calculate the theoretical concentration of the spiked sample:

$$\text{heoretical concentration} = \frac{(C_u \times V_u) + (C_s \times V_s)}{V_u + V_s}$$

Where:

C_u = measured concentration of sample, in mg/L (µg/L divided by 1000)

V_u = volume of sample in mL

C_s = concentration of chlorine standard (mg/L, certificate value)

V_s = volume of standard added in mL

6. Calculate the percent spiked recovery:

$$\text{Spike Recovery} = \frac{\text{Spiked sample result, in mg/L}}{\text{Theoretical concentration calculated, in mg/L}} \times 100$$

Example:

Sample result (C_u) = 120 µg/L or 0.120 mg/L

Spiked sample result = 185 µg/L or 0.185 mg/L

Volume Sample (V_u) = 200 mL

Volume Standard (V_s) = 0.2 mL

Chlorine Standard (C_s) = 68.1 mg/L

* The standard additions technique is not applicable for samples containing excess reducing agents such as sulfur dioxide, sulfite, or bisulfite.

$$\text{heoretical concentration} = \frac{(0.120 \times 200) + (68.1 \times 0.2)}{200 + 0.2} = 0.188 \text{ mg/L}$$

Ideally, the percent recovery should be 100%. Generally, results from 80-120% recovery are considered acceptable.

Precision

In a single laboratory, using a standard solution of 347 µg/L chlorine, a single operator obtained a standard deviation of ± 3.2 µg/L chlorine.

Detection Limit

With good operator technique, the estimated detectable concentration is approximately 15 µg/L chlorine using 0.00564 N PAO.

Sampling and Storage

Chlorine is rapidly lost from water. Avoid exposure to sunlight or other strong light. Avoid excessive agitation. Analyze samples immediately.

Interferences

- Silver ions poison the electrode.
- Copper ions interfere.
- Interferences are sometimes found in highly turbid water and those containing surface active agents. Oxidized manganese and other oxidizing reagents give positive interferences.
- Some uncertainty in the end point may be observed with samples containing high organic content.
- Samples containing excess reducing agents, such as sulfur dioxide, sulfite, and bisulfite, do not contain free chlorine or chloramines and can not be titrated under the conditions of the test.
- Highly buffered samples or extreme sample pH may exceed the buffering capacity of the buffer reagent. If necessary, add additional buffer and check pH of sample prior to titration.

Summary of Method

In the amperometric forward titration procedure for total chlorine, a small electrical current is applied across two identical platinum electrodes. No current can flow between the electrodes unless a substance that can be oxidized at the anode and a substance that can be reduced at the cathode are both present. In the case of the total chlorine, an equivalent amount of iodine forms from the reaction of excess iodide with chlorine and combined chlorine at pH 4. During the titration with phenylarsine oxide (PAO), the free iodine is reduced to iodide at the cathode and PAO is oxidized from the +3 oxidation state to the +5 oxidation state at the anode. Prior to the end point of the titration, both iodine and iodide are present in solution; therefore current can flow, even with a very small applied potential. At the end point, no free iodine remains and the solution cannot conduct even if excess PAO titrant is added. The end point is defined when no change in current occurs, signaling all total chlorine has been reacted.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Phenylarsine Oxide Solution 0.00564 N Digital Titrator cartridge	each	1999-01
Acetate Buffer Solution, pH 4	100 mL MBD	14909-32
Potassium Iodide Powder Pillows	100/pkg	1077-99

REQUIRED APPARATUS

Amperometric Titrator Assembly	each	19299-00
Digital Titrator	each	16900-01
Beaker, low-form, 250-mL	each	500-46
Cylinder, graduated, 250-mL	each	508-46
Delivery Tubes, 90° with hook	5/pkg	41578-00
Probe Assembly, Amperometric Titrator	each	19390-00
Stir Bar, octagonal, Teflon-coated, 50.8 x 7.9 mm	each	20953-55
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

OPTIONAL REAGENTS

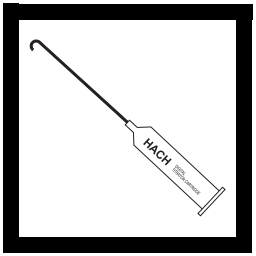
Chlorine Standard Solution PourRite™ Ampules, 50-75 mg/L Cl_2, 2-mL	20/pkg	14268-20
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

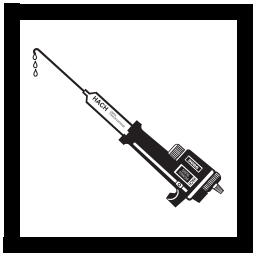
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette® Pipet	50/pkg	21856-96
PourRite™ Ampule Breaker	each	24846-00
Standard Methods for the Examination of Water and Wastewater, 19th edition	each	22708-00

CHROMATE (20 to > 400 mg/L as CrO_4^{2-})

Using Sodium Thiosulfate

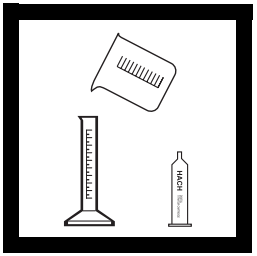


1. Insert a clean delivery tube into the Sodium Thiosulfate titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step,* for assembly instructions, if necessary.



2. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

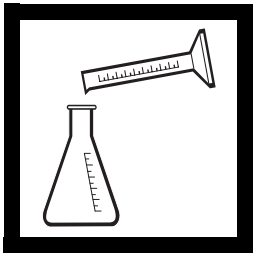
Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*



3. Select a sample volume corresponding to the expected chromate (CrO_4^{2-}) concentration from *Table 1*.

Note: *Collect 200 to 300 mL of sample in an acid-washed glass or polyethylene container.*

Note: *See Sampling and Storage following these steps.*



4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample to a clean 125-mL Erlenmeyer flask. Dilute to about the 50-mL mark with deionized water.

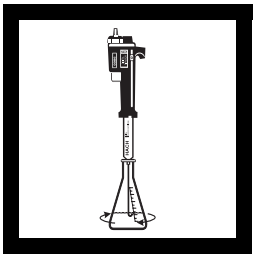
Table 1

Range (mg/L as CrO_4^{2-})	Sample Volume (mL)	Titration Cartridge (N $Na_2S_2O_3$)	Catalog Number	Digit Multiplier
20-80	50	0.2068 N	22676-01	0.2
50-200	20	0.2068 N	22676-01	0.5
100-400	10	0.2068 N	22676-01	1.0
> 400	5	0.2068 N	22676-01	2.0

5. Add the contents of one Potassium Iodide Powder Pillow and swirl to mix.

6. Add the contents of one Dissolved Oxygen 3 Reagent Powder Pillow and swirl to mix. Wait at least three minutes but not more than 10 minutes before completing *steps 7 to 9.*

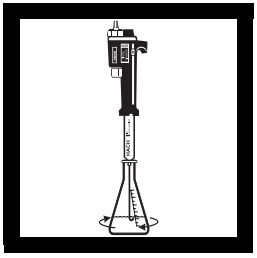
Note: *A yellow or brown color indicates the presence of chromate.*



7. Place the delivery tube tip into the solution and swirl the flask while titrating with sodium thiosulfate to a straw-yellow color.

8. Add one dropper of Starch Indicator Solution and swirl to mix.

Note: *A blue color will form.*



9. Continue titrating until the solution turns from blue to colorless. Record the number of digits required.

10. Calculate:

Total Digits Required x Digit Multiplier = mg/L chromate (CrO_4^{2-})

Note: *Results may be expressed as mg/L sodium chromate (Na_2CrO_4) or chromium (Cr) by multiplying the mg/L chromate by 1.4 or 0.448, respectively.*

Sampling and Storage

Collect 200 to 300 mL of sample in an acid-washed glass or polyethylene container. If sample cannot be analyzed immediately add 1 mL concentrated sulfuric acid and swirl to mix.

Accuracy Check

Standard Additions Method

This accuracy check should be performed when interferences are suspected or to verify analytical technique.

1. Use a TenSette® Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of Hexavalent Chromium Standard Solution, 1000 mg/L to three samples of the same volume as that titrated in the procedure.

2. Analyze each as described in the procedure.

3. Each 0.1 mL addition of standard should require 22 additional digits of titrant. If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

Standard Preparation

A standard solution equivalent to 67 mg/L chromate (30 mg/L Cr) can be prepared by diluting 3.0 mL of Hexavalent Chromium Standard Solution, 1000 mg/L Cr to 100 mL in a volumetric flask. Titrate a 20-mL or 50-mL sample as described in the procedure.

Interferences

Substances capable of oxidizing iodide to iodine under acidic conditions (such as ferric iron and copper) will interfere to give high results. The effects of iron and copper may be masked by dissolving a Magnesium CDTA Powder Pillow, followed by two 1.0-gram measuring spoons of Sodium Acetate in the sample between *steps 6 and 7*.

Summary of Method

Chromate in the sample reacts with iodide under acidic conditions to form iodine as triiodide. Addition of starch indicator produces a blue color complex with the iodine. This complex is titrated with sodium thiosulfate to a colorless end point. The volume of titrant used is proportional to the chromate concentration.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Chromate Reagent Set (about 100 tests)		22724-00
Includes: (1) 349-32, (1) 987-99, (1) 20599-96, (1) 22676-01		
Dissolved Oxygen 3 Reagent Powder Pillows	100/pkg	987-99
Potassium Iodide Powder Pillows	50/pkg	20599-96
Sodium Thiosulfate Titration Cartridge, 0.2068 N	each	22676-01
Starch Indicator Solution	100 mL MDB	349-32
Water, deionized	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening pillows	each	968-00
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 125-mL	each	505-43
Select one or more based on sample concentration:		
Cylinder, graduated, 10-mL	each	508-38
Cylinder, graduated, 25-mL	each	508-40
Cylinder, graduated, 50-mL	each	508-41

OPTIONAL REAGENTS

Chromium, Hexavalent, Standard Solution, 1000 mg/L	100 mL	14664-42
Magnesium CDTA Powder Pillows	100/pkg	14080-99
Sodium Acetate, trihydrate, ACS	100 g	178-26

OPTIONAL APPARATUS

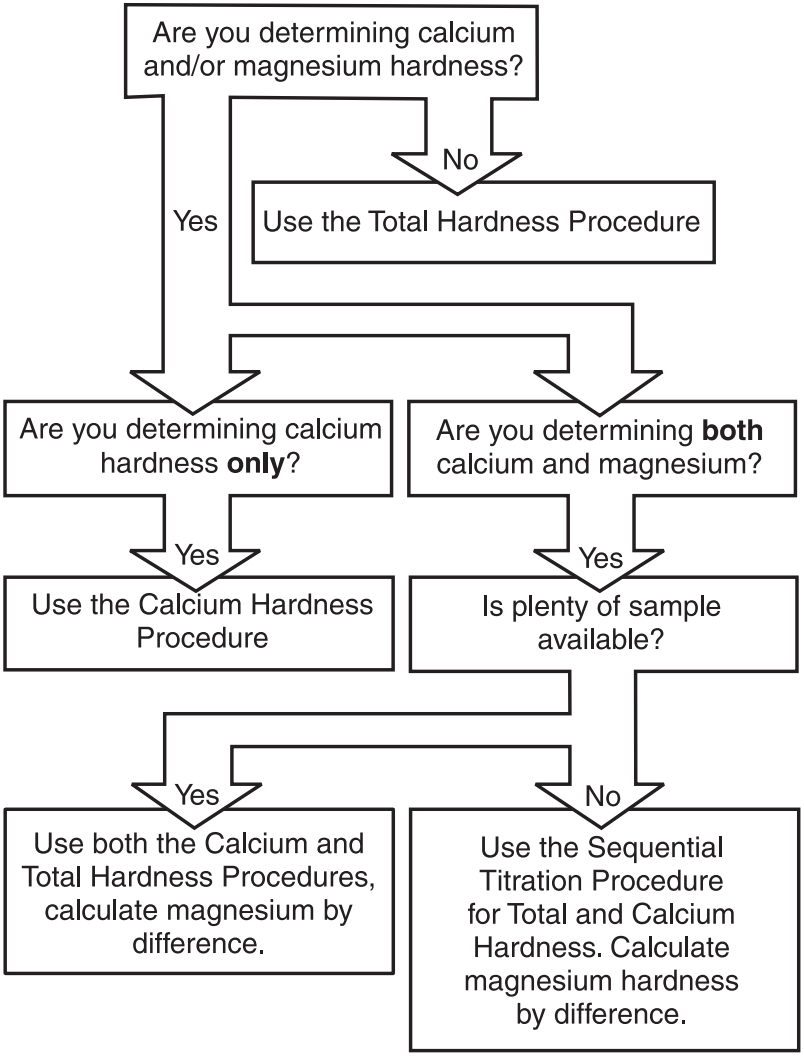
Clamp, 2-prong, extension	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473-mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook for TitraStir® Stir Plate	5/pkg	41578-00
Flask, volumetric, Class B, 100 mL	each	547-42
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01

OPTIONAL APPARATUS, continued

Description	Unit	Cat. No.
Pipet Tips for 19700-01 TenSette® Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 3-mL	each	14515-03
Pipet, volumetric, Class A, 5-mL	each	14515-37
Pipet, volumetric, Class A, 10-mL	each	14515-38
Pipet, volumetric, Class A, 20-mL	each	14515-20
Pipet, volumetric, Class A, 50-mL	each	14515-41
Pipet Filler, safety bulb	each	14651-00
Spoon, measuring, 1.0-gram	each	510-00
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

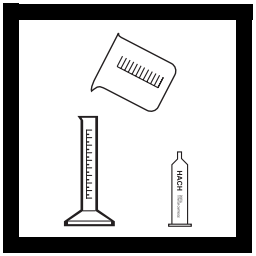
HARDNESS DECISION TREE

There are several hardness procedures presented in this manual. Use the following decision tree to select the appropriate procedure for your application.



HARDNESS, CALCIUM (10 to 4000 mg/L as $CaCO_3$)

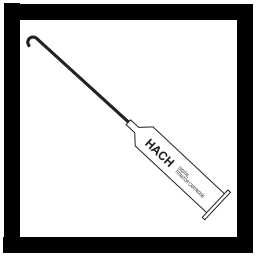
Using EDTA



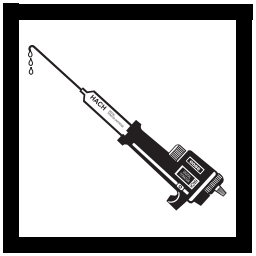
1. Select a sample size and an EDTA Titration Cartridge corresponding to the expected calcium as calcium carbonate ($CaCO_3$) concentration. Use *Table 1* for concentrations in mg/L or *Table 2* for concentrations in German degrees of hardness (G.d.h.).

Note: *One German degree hardness equals 17.9 mg/L hardness as $CaCO_3$.*

Note: *If sample cannot be analyzed immediately, add 1.5 mL Nitric Acid per liter of sample to preserve the sample and to prevent adsorption of the calcium to the container walls. Store in a refrigerator. Samples preserved in this manner are stable for one week. Neutralize to pH 7 before running the test.*



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step* for assembly instructions, if necessary.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1* or *Table 2*. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

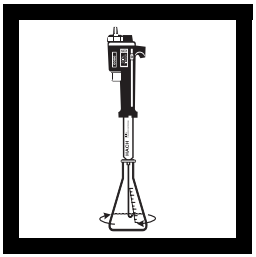
5. Add 2 mL of 8 N Potassium Hydroxide Standard Solution and swirl to mix.

Note: *For samples of 50 mL or less, 1 mL may be added.*

Note: *Magnesium is not included in the results but must be present for a sharp end point. If it is known to be absent, add one to two drops of Magnesium Standard Solution, 10 g/L as $CaCO_3$.*

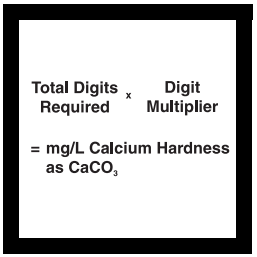
6. Add the contents of one CalVer® 2 Calcium Indicator Powder Pillow (Cat. No. 852-99) and swirl to mix.

Note: *A 0.1-gram scoop of CalVer 2 Calcium Indicator Powder (Cat. No. 281-14) may be substituted here.*



7. Place the delivery tube tip into the solution and swirl the flask while titrating with EDTA from pink to blue. Record the number of digits required.

Note: *Titrate slowly near the end point, because the reaction is slow, especially in cold samples.*



8. Calculate the sample concentration using one of the formulas below:

Total Digits Required x Digit Multiplier (*Table 1*) = mg/L Calcium Hardness as $CaCO_3$

Total Digits Required x Digit Multiplier (*Table 2*) = G.d.h.

Table 1

Range (mg/L as $CaCO_3$)	Sample Volume (mL)	Titration Cartridge (M EDTA)	Catalog Number	Digit Multiplier
10-40	100	0.0800	14364-01	0.1
40-160	25	0.0800	14364-01	0.4
100-400	100	0.800	14399-01	1.0
200-800	50	0.800	14399-01	2.0
500-2000	20	0.800	14399-01	5.0
1000-4000	10	0.800	14399-01	10.0

Table 2

Range (G.d.h.)	Sample Volume (mL)	Titration Cartridge (M EDTA)	Catalog Number	Digit Multiplier
1-4	100	0.1428	14960-01	0.01
4-16	25	0.1428	14960-01	0.04
10-40	50	0.714	14959-01	0.1
25-100	20	0.714	14959-01	0.25
>100	10	0.714	14959-01	0.5

Hardness Relationships

mg/L Ca = Ca Hardness, mg/L as $CaCO_3$ x 0.40

Accuracy Check

Standard Additions Method
This accuracy check should be performed when interferences are suspected or to verify analytical technique.

1. Snap the neck off a Hardness Standard Solution Voluette® Ampule, 10,000 mg/L as $CaCO_3$.
2. Use a TenSette® Pipet to add 0.1 mL of standard to the solution titrated in *step 7*. Resume titration back to the same end point. Record the number of digits required.
3. Repeat, using two more additions of 0.1 mL. Titrate to the end point after each addition.
4. Each 0.1 mL addition of standard should require 10 additional digits of 0.800 N titrant or 100 digits of 0.0800 N titrant (11 digits of 0.714 M or 56 digits of 0.1428 M titrant). If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

Interferences

WARNING:
Potassium cyanide is toxic. Always add it after the potassium hydroxide. Excess potassium cyanide does not affect results. All cyanide wastes should be disposed of by adding an excess of strongly alkaline sodium hypochlorite solution (bleach) with stirring. Use good ventilation. Allow to stand for 24 hours before disposal.

- Some transition and heavy metals complex the indicators and prevent the color change at the end point. Adding a 0.5-g scoop of potassium cyanide (KCN) after the addition of potassium hydroxide removes interference from the following metals at the levels listed (in an undiluted 100-mL sample), see *Table 3*.

Table 3

Metal	Max. Tolerance Level* with KCN	Max. Tolerance Level* without KCN present
Cobalt	20 mg/L	none
Copper	100 mg/L	0.10 mg/L
Nickel	200 mg/L	0.5 mg/L
Zinc	100 mg/L	5 mg/L

* Proportionally higher levels of these elements are tolerable in smaller sample sizes since their effect is diluted when bringing the volume to 100 mL. Because Tables 1 and 2 have sample volumes of 10-100 mL, the interference concentrations may be greater in your sample and have no effect because of sample dilution.

- Iron interferes above 8 mg/L in undiluted samples. Above this level, it causes a red-orange to green end point which is sharp and usable up to 20 mg/L iron.
- Manganese interferes above 5 mg/L.
- Aluminum causes a slow end point, but up to 200 mg/L can be tolerated by allowing enough time for color change.
- Magnesium interference up to 200 mg/L is prevented by formation of magnesium hydroxide at the high test pH, but higher levels prevent a distinct end point.
- Orthophosphate causes a slow end point, but does not interfere if the calcium phosphate that forms is allowed enough time to redissolve during the titration. Polyphosphate must be absent for accurate results.
- Barium and strontium are titrated with calcium but seldom present in natural waters in significant amounts.
- Acidity and alkalinity at 10,000 mg/L as $CaCO_3$ do not interfere.
- Saturated sodium chloride solutions do not give a distinct end point, but the titration can be run directly on sea water.

- Samples at about 20 °C (68 °F) or colder should be titrated slowly near the end point to allow enough time for the color change.
- Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.

Summary of Method

The sample is made alkaline (pH 12-13) with potassium hydroxide to precipitate magnesium as magnesium hydroxide. CalVer 2 Indicator is added and combines with any calcium to form a pink-red color. As EDTA is added, it reacts with the free calcium ions present. When no free calcium ions remain, the EDTA then removes the calcium complexed with the indicator, causing a color change to blue.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Calcium Hardness Reagent Sets (about 100 tests)		
1-16 G.d.h. includes: (1) 282-32, (1) 852-99, (1) 14960-01		24473-00
10-100+ G.d.h. includes: (1) 282-32, (1) 852-99, (1) 14959-01		24474-00
10-160 mg/L includes: (1) 282-32, (1) 852-99, (1) 14364-01		24472-00
100-4,000 mg/L includes: (1) 282-32, (1) 852-99, (1) 14399-01		24475-00
CalVer® 2 Indicator Powder Pillows	100/pkg	852-99
Potassium Hydroxide Standard Solution, 8.00 N	100 mL MDB*	282-32
Water, deionized	4 L	272-56
Select one or more based on sample concentration:		
EDTA Titration Cartridge, 0.0800 M	each	14364-01
EDTA Titration Cartridge, 0.1428 M	each	14960-01
EDTA Titration Cartridge, 0.714 M	each	14959-01
EDTA Titration Cartridge, 0.800 M	each	14399-01

REQUIRED APPARATUS

Digital Titrator	each	16900-01
Flask, Erlenmeyer, 250 mL	each	505-46
Select one or more based on sample concentration:		
Cylinder, graduated, 10 mL	each	508-38
Cylinder, graduated, 25 mL	each	508-40
Cylinder, graduated, 50 mL	each	508-41
Cylinder, graduated, 100 mL	each	508-42

* Marked Dropper Bottle (MDB). Contact Hach for larger sizes.

OPTIONAL REAGENTS

Calcium and Magnesium Total Hardness Reagent Set (about 100 tests) 22721-00
Includes: (2) 282-32, (1) 424-32, (1) 851-99, (1) 947-99, (1) 14364-01, (1) 14399-01

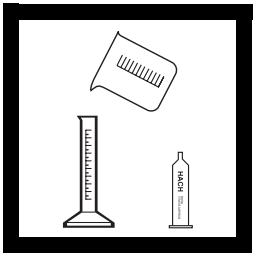
Description	Unit	Cat. No.
Calcium Chloride Standard Solution, 1000 mg/L as $CaCO_3$	1000 mL	121-53
CalVer® 2 Calcium Indicator Powder	113 g	281-14
Calcium Standard Solution Voluette® Ampules, 10,000 mg/L as $CaCO_3$, 10-mL	16/pkg	2187-10
Magnesium Standard Solution, 10 g/L $CaCO_3$	29 mL SCDB	1022-33
Nitric Acid, ACS	500 mL	152-49
Nitric Acid Solution, 1:1	500 mL	2540-49
Potassium Cyanide, ACS	125 g	767-14

OPTIONAL APPARATUS

Bottle, wash, poly, 500-mL	each	620-11
Clamp, 2-prong, extension, 38-mm	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473 mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook	5/pkg	41578-00
pH Paper, 1.0 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, volumetric, Class A, 10-mL	each	14515-38
Pipet, volumetric, Class A, 20-mL	each	14515-20
Pipet, volumetric, Class A, 25-mL	each	14515-40
Pipet, volumetric, Class A, 50-mL	each	14515-41
Pipet, volumetric, Class A, 100-mL	each	14515-42
Pipet Filler, safety bulb	each	14651-00
*sens**ion**™1* Basic Portable pH Meter with electrode	each	51700-10
Spoon, measuring, 0.1-gram	each	511-00
Spoon, measuring, 0.5-gram	each	907-00
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10
Voluette® Ampule Breaker Kit	each	21968-00

HARDNESS, TOTAL (10 to 4000 mg/L as $CaCO_3$)

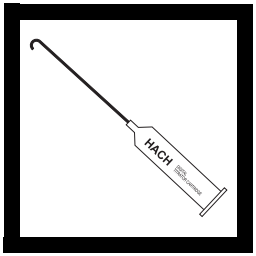
Using EDTA



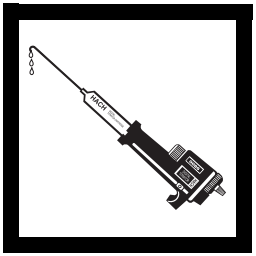
1. Select a sample size and an EDTA Titration Cartridge corresponding to the expected total hardness as calcium carbonate ($CaCO_3$) concentration. Use *Table 1* for concentrations in mg/L or *Table 2* for concentrations in German degrees of hardness (G.d.h.).

Note: *One German degree hardness equals 17.9 mg/L hardness as $CaCO_3$.*

Note: *Collect at least 100 mL of sample in a glass or polyethylene container. Samples may be held up to seven days before analysis if stored at 4 °C and acidified to pH 2 with concentrated nitric acid. Neutralize acidified sample to pH 7 with ammonium hydroxide before testing.*



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step* for assembly instructions, if necessary.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

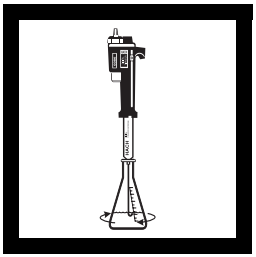
Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1* or *Table 2*. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

5. Add 2 mL of Hardness 1 Buffer Solution and swirl to mix.

6. Add the contents of one ManVer® 2 Hardness Indicator Powder Pillow (Cat. No. 851-99) and swirl to mix.

Note: *Four drops of ManVer Hardness Indicator Solution or a 0.1 g scoop of ManVer 2 Hardness Indicator Powder (Cat. No. 280-14) may be substituted for the powder pillow.*



7. Place the delivery tube tip into the solution and swirl the flask while titrating with EDTA from red to pure blue. Record the number of digits required.

Note: *Titrate slowly near the end point because the reaction is slow, especially in cold samples.*

8. Use one of the following formulas to calculate the final concentration:

Digits Required x Digit Multiplier (*Table 1*) = mg/L Total Hardness as $CaCO_3$

Digits Required x Digit Multiplier (*Table 2*) = G.d.h.

Note: *The magnesium concentration may be determined by subtracting the results of the calcium determination from total hardness.*

Table 1

Range (mg/L as $CaCO_3$)	Sample Volume (mL)	Titration Cartridge (M EDTA)	Catalog Number	Digit Multiplier
10-40	100	0.0800	14364-01	0.1
40-160	25	0.0800	14364-01	0.4
100-400	100	0.800	14399-01	1.0
200-800	50	0.800	14399-01	2.0
500-2000	20	0.800	14399-01	5.0
1000-4000	10	0.800	14399-01	10.0

Table 2

Range (G.d.h.)	Sample Volume (mL)	Titration Cartridge (M EDTA)	Catalog Number	Digit Multiplier
1-4	100	0.1428	14960-01	0.01
4-16	25	0.1428	14960-01	0.04
10-40	50	0.714	14959-01	0.1
25-100	20	0.714	14959-01	0.25
>100	10	0.714	14959-01	0.5

Hardness Relationships

$$\text{g/L Total Hardness as Ca} = \text{mg/L Total Hardness as } (CaCO_3) \times 0.400$$

$$\text{mg/L Total Hardness (as } CaCO_3) = \text{mg/L Ca (as } CaCO_3) + \text{mg/L Mg (as } CaCO_3)$$

Accuracy Check

Standard Additions Method
To verify analytical technique, use 20 mL of the Calcium Standard Solution, 1000 mg/L as $CaCO_3$. Perform the procedure as described above. This solution will read 1000 mg/L or 55.9 G.d.h.

Perform this accuracy check when interferences are suspected.

1. Snap the neck off a Hardness Standard Solution Voluette® Ampule, 10,000 mg/L as $CaCO_3$.
2. Use a TenSette® Pipet to add 0.1 mL of standard to the sample titrated in *step 7*. Resume titration back to the same end point. Record the number of digits required.
3. Repeat, using two more additions of 0.1 mL. Titrate to the end point after each addition.
4. Each 0.1 mL addition of standard should require 10 additional digits of 0.800 M titrant, 100 digits of 0.0800 M titrant, 11 digits of 0.714 M, or 56 digits of 0.1428 M titrant. If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

Interferences

- Although less common than calcium and magnesium, other polyvalent metal ions cause the same hardness effects and will be included in the results.
- Some transition and heavy metals complex the indicator and prevent the color change at the end point.
- Iron does not interfere up to 15 mg/L. Above this level it causes a red-orange to green end point which is sharp and

usable up to 30 mg/L iron. Substitute a 0.0800 M CDTA or 0.800 M CDTA titration cartridge for the 0.0800 M EDTA or 0.800 M EDTA titration cartridges, respectively, if iron interference is probable.

- Manganese titrates directly up to 20 mg/L but masks the end point above this level. Adding a 0.1-gram scoop of hydroxylamine hydrochloride monohydrate raises this level to 200 mg/L manganese.

WARNING
Potassium cyanide is toxic. Always add it after the potassium hydroxide. Excess potassium cyanide does not affect results. All cyanide wastes should be disposed of by adding an excess of strongly alkaline sodium hypochlorite solution (bleach) with stirring. Use good ventilation. Allow to stand for 24 hours before disposal.

- Copper and aluminum interfere at levels above 0.10 and 0.20 mg/L, respectively. Cobalt and nickel interfere at all levels and must be absent or masked. A 0.5-gram scoop of potassium cyanide removes interference from up to 100 mg/L copper, 100 mg/L zinc, 100 mg/L cobalt, and 100 mg/L nickel. It raises the permissible aluminum level to 1 mg/L. Metals masked with cyanide will not be included in the hardness result.
- Orthophosphate causes a slow end point and polyphosphate must be absent for accurate results.
- Acidity and alkalinity at 10,000 mg/L (as $CaCO_3$) do not interfere.
- Saturated sodium chloride solutions do not give a distinct end point, but the titration can be run directly on sea water.
- Adding the contents of one CDTA Magnesium Salt Powder Pillow removes metal interferences at or below the levels shown in *Table 3*.

Table 3

Metal	CDTA Removes Interference Below This Level
Aluminum	50 mg/L
Cobalt	200 mg/L
Copper	100 mg/L
Iron	100 mg/L
Manganese	200 mg/L
Nickel	400 mg/L
Zinc	300 mg/L

- If more than one metal is present at or above the concentrations shown above, an additional CDTA Magnesium Salt Powder Pillow may be required.

Results obtained by this procedure include the hardness contributed by the metals. If the concentration of each metal is known, a correction can be applied to obtain the calcium and magnesium hardness concentration. The hardness (in mg/L as $CaCO_3$) contributed by each mg/L of metal is listed below, and can be subtracted from the total hardness value obtained above to determine the calcium and magnesium hardness. See *Table 4*.

Table 4

Metal	Hardness as $CaCO_3$ Contributed by Each mg/L of Metal
Aluminum	3.710
Barium	0.729
Cobalt	1.698
Copper	1.575
Iron	1.792
Manganese	1.822
Nickel	1.705
Strontium	1.142
Zinc	1.531

- Barium, strontium and zinc titrate directly.
- Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment.

Summary of Method

After the sample is buffered to pH 10.1, ManVer 2 Hardness Indicator is added, and forms a red complex with a portion of the calcium and magnesium in the sample. EDTA titrant reacts first with the free calcium and magnesium ions, then with those bound to the indicator, causing it to change to a blue color at the end point.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Total Hardness Reagent Sets (about 100 tests)		
1-16 G.d.h. includes: (1) 424-32, (1) 851-99, (1) 14960-01		24478-00
10-100+ G.d.h. includes: (1) 424-32, (1) 851-99, (1) 14959-01		24479-00
10-160 mg/L includes: (1) 424-32, (1) 851-99, (1) 14364-01		24480-00
100-4,000 mg/L includes: (1) 424-32, (1) 851-99, (1) 14399-01		24481-00
Hardness 1 Buffer Solution	100 mL MDB	424-32
ManVer® 2 Hardness Indicator Powder Pillow	100/pkg	851-99
Water, deionized	4 L	272-56
Select one or more based on sample concentration:		
EDTA Titration Cartridge, 0.0800 M	each	14364-01
EDTA Titration Cartridge, 0.1428 M	each	14960-01
EDTA Titration Cartridge, 0.714 M	each	14959-01
EDTA Titration Cartridge, 0.800 M	each	14399-01

REQUIRED APPARATUS

Digital Titrator	each	16900-01
Flask, Erlenmeyer, 250-mL	each	505-46
Select one or more based on sample concentration:		
Cylinder, graduated, 10-mL	each	508-38
Cylinder, graduated, 25-mL	each	508-40
Cylinder, graduated, 50-mL	each	508-41
Cylinder, graduated, 100-mL	each	508-42

OPTIONAL REAGENTS

Ammonium Hydroxide, 10%	100 mL MDB*	14736-32
Calcium Chloride Standard Solution, 1000 mg/L as $CaCO_3$	1000 mL	121-53
CDTA Magnesium Salt Powder Pillows	100/pkg	14080-99
CDTA Titration Cartridge, 0.0800 M	each	14402-01
CDTA Titration Cartridge, 0.800 M	each	14403-01
Calcium Standard Solution Voluette® Ampules, 10,000 mg/L as $CaCO_3$, 10-mL	16/pkg	2187-10
Hydroxylamine Hydrochloride, Monohydrate, ACS	113 g	246-14
ManVer® 2 Hardness Indicator Powder	113 g	280-14
ManVer® Hardness Indicator Solution	100 mL MDB*	425-32
Nitric Acid Solution, 1:1	500 mL	2540-49
Nitric Acid, ACS	500 mL	152-49
Potassium Cyanide, ACS	125 g	767-14

* Contact Hach for larger sizes.

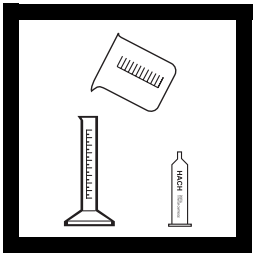
OPTIONAL APPARATUS

Description	Unit	Cat. No.
Bottle, wash, poly, 500-mL	each	620-11
Clamp 2-prong, extension, 38-mm	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473-mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook	5/pkg	41578-00
pH Paper, 1.0 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, volumetric, Class A, 10-mL	each	14515-38
Pipet, volumetric, Class A, 20-mL	each	14515-20
Pipet, volumetric, Class A, 25-mL	each	14515-40
Pipet, volumetric, Class A, 50-mL	each	14515-41
Pipet, volumetric, Class A, 100-mL	each	14515-42
Pipet Filler, safety bulb	each	14651-00
*sens**ion***™*1* Basic Portable pH Meter with electrode	each	51700-10
Spoon, measuring, 0.1-gram	each	511-00
Spoon, measuring, 0.5-gram	each	907-00
Spoon, measuring, 1.0-gram	each	510-00
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10
Voluette® Ampule Breaker Kit	each	21968-00

HARDNESS, TOTAL, SEQUENTIAL (10 to 4000 mg/L as $CaCO_3$)

Sequential Titration Procedure (Limited Sample)

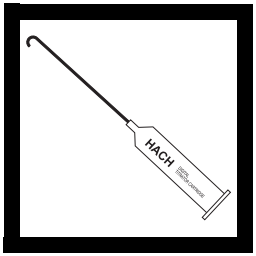
Scope and Application: To determine total and calcium hardness in samples with limited sample size, follow this procedure. Calculate magnesium hardness by difference.



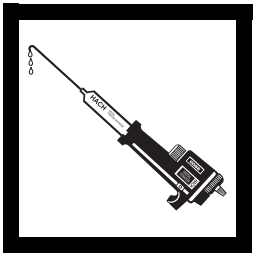
1. Select a sample size and an EDTA Titration Cartridge corresponding to the expected calcium as calcium carbonate ($CaCO_3$) concentration. Use *Table 1* for concentrations in mg/L or *Table 2* for concentrations in German degrees of hardness (G.d.h.).

Note: *One German degree hardness equals 17.9 mg/L hardness as $CaCO_3$.*

Note: *If sample cannot be analyzed immediately, add 1.5 mL Nitric Acid per liter of sample to preserve the sample and to prevent adsorption of the calcium to the container walls. Store in a refrigerator. Samples preserved in this manner are stable for one week. Neutralize to pH 7 before running the test.*



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step* for assembly instructions, if necessary.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience, use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1* or *Table 2*. Transfer the sample into a clean 250-mL Erlenmeyer flask. Dilute to about the 100-mL mark with deionized water, if necessary.

Table 1

Range (mg/L as $CaCO_3$)	Sample Volume (mL)	Titration Cartridge (M EDTA)	Catalog Number	Digit Multiplier
10-40	100	0.0800	14364-01	0.1
40-160	25	0.0800	14364-01	0.4
100-400	100	0.800	14399-01	1.0
200-800	50	0.800	14399-01	2.0
500-2000	20	0.800	14399-01	5.0
1000-4000	10	0.800	14399-01	10.0

Table 2

Range (G.d.h.)	Sample Volume (mL)	Titration Cartridge (M EDTA)	Catalog Number	Digit Multiplier
1-4	100	0.1428	14960-01	0.01
4-16	25	0.1428	14960-01	0.04
10-40	50	0.714	14959-01	0.1
25-100	20	0.714	14959-01	0.25
>100	10	0.714	14959-01	0.5

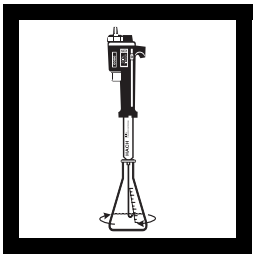
5. Add 2 mL of 8 N Potassium Hydroxide Standard Solution and swirl to mix.

Note: *For samples of 50 mL or less, 1 mL may be added.*

Note: *Magnesium is not included in the results but must be present for a sharp end point. If it is known to be absent, add 1–2 drops of Hardness Standard Solution.*

6. Add the contents of one CalVer® 2 Calcium Indicator Powder Pillow (Cat. No. 947-99) and swirl to mix.

Note: *Do not use potassium cyanide to eliminate interferences or toxic gas will form in subsequent steps.*



7. Place the delivery tube tip into the solution and swirl the flask while titrating with EDTA from pink to blue. Record the number of digits required.

Note: *Titrate slowly near the end point, because the reaction is slow, especially in colder samples.*

8. Calculate the sample concentration of calcium hardness by using one of the formulas below:

Digits Required x Digit Multiplier (*Table 1*) = mg/L Calcium Hardness as CaCO3

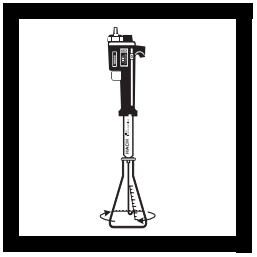
Digits Required x Digit Multiplier (*Table 2*) = G.d.h.

Note: *Do not reset the counter to zero.*

9. After completing the calcium titration, add 1 mL of 5.25 N Sulfuric Acid Standard Solution. Add additional acid dropwise and swirl the solution until the color changes from pure blue to purple, then to blue and finally to red. Swirl the flask to ensure that all precipitated magnesium hydroxide has re-dissolved.

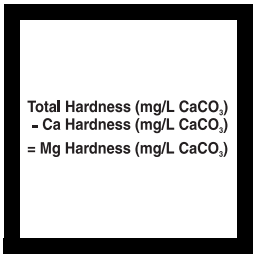
10. Add 2 mL of Hardness 1 Buffer Solution and swirl to mix.

11. Add the contents of one ManVer® 2 Hardness Indicator Powder Pillow (Cat. No. 928-99) or 4 drops of Hardness 2 Test Solution (Cat. No. 425-32). Swirl to mix.



12. Place the delivery tube tip into the solution and swirl the flask while titrating with EDTA from red to pure blue. Record the number of digits required.

Note: *Titrate slowly near the end point because the reaction is slow, especially in colder samples.*



13. Use the appropriate formula below to calculate the final concentration based on sample size and cartridge used:

Digits Required x Digit Multiplier (*Table 1*) = mg/L Total Hardness as $CaCO_3$

Digits Required x Digit Multiplier (*Table 2*) = G.d.h.

14. The first titration gives the mg/L calcium hardness and the second gives the mg/L total hardness. The difference between the values is the mg/L magnesium hardness as $CaCO_3$.

Total Hardness (mg/L $CaCO_3$) - Ca Hardness (mg/L $CaCO_3$) = Mg Hardness (mg/L $CaCO_3$)

Note: *See below for conversion factors.*

Hardness Relationships

mg/L Mg Hardness as $CaCO_3$
= mg/L Total Hardness as $CaCO_3$ – mg/L Ca Hardness as $CaCO_3$

mg/L $MgCO_3$ = mg/L Mg Hardness as $CaCO_3 \times 0.842$

mg/L Mg = mg/L $MgCO_3 \times 0.29$

Interferences

WARNING:
Do not use potassium cyanide to eliminate interferences because it will generate deadly hydrogen cyanide gas when the sulfuric acid solution is added in step 9.

- Although less common than calcium and magnesium, other polyvalent metal ions cause the same hardness effects and will be included in the results.

- Some transition and heavy metals complex the indicator and prevent the color change at the end point.
- Iron does not interfere up to 15 mg/L. Above this level it causes a red-orange to green end point which is sharp and usable up to 30 mg/L iron. Substitute a 0.0800 M CDTA or 0.800 M CDTA titration cartridge for the 0.0800 M EDTA or 0.800 M EDTA titration cartridges, respectively, if iron interference is probable. For results in G.d.h., divide the mg/L result by 17.9.
- Manganese titrates directly up to 20 mg/L but masks the end point above this level. Adding a 0.1-gram scoop of hydroxylamine hydrochloride raises this level to 200 mg/L manganese.
- Copper interferes at levels of 0.10 and 0.20 mg/L. Cobalt and nickel interfere at all levels and must be absent or masked.
- Orthophosphate causes a slow end point and polyphosphate must be absent for accurate results.
- Acidity and alkalinity at 10,000 mg/L (as $CaCO_3$) do not interfere.
- Saturated sodium chloride solutions do not give a distinct end point, but the titration can be run directly on sea water.
- Adding the contents of one CDTA Magnesium Salt Powder Pillow removes metal interferences at or below the levels shown in *Table 3*.
- If more than one metal is present at or above the concentrations shown in *Table 3*, an additional CDTA Magnesium Salt Powder Pillow may be required.
- Results obtained by this procedure include the hardness contributed by polyvalent metal ions. If the concentration of each metal is known, a correction can be applied to obtain the calcium and magnesium hardness concentration. The hardness (in mg/L as $CaCO_3$) contributed by each mg/L of metal is listed in *Table 4*. Hardness contributed by metals can be subtracted from the total hardness value obtained in

step 13 to determine the calcium and magnesium hardness concentration.

- Barium, strontium and zinc titrate directly.

Table 3

Metal	CDTA Removes Interference Below this Level
Aluminum	50 mg/L
Cobalt	200 mg/L
Copper	100 mg/L
Iron	100 mg/L
Manganese	200 mg/L
Nickel	400 mg/L
Zinc	300 mg/L

Table 4

Metal	Hardness as $CaCO_3$ Contributed by Each mg/L of Metal
Aluminum	3.710
Barium	0.729
Cobalt	1.698
Copper	1.575
Iron	1.792
Manganese	1.822
Nickel	1.705
Strontium	1.142
Zinc	1.531

REQUIRED REAGENTS*

Calcium and Magnesium Hardness Reagent Sets (about 100 tests each)

10-100+ G.d.h. includes: (1) 282-32, (1) 424-32, (1) 928-99, (1) 947-99, (1) 2449-32, (1) 14959-01 24485-00

10-160 mg/L includes: (1) 282-32, (1) 424-32, (1) 928-99, (1) 947-99, (1) 2449-32, (1) 14364-01 24486-00

100-4,000 mg/L includes: 282-32, (1) 424-32, (1) 928-99, (1) 947-99, (1) 2449-32, (1) 14399-01 24487-00

Description	Unit	Cat. No.
CalVer® 2 Indicator Powder Pillows	100/pkg	947-99
Hardness 1 Buffer Solution	100 mL MDB	424-32
ManVer® 2 Hardness Indicator Powder Pillows	100/pkg	928-99
Potassium Hydroxide Standard Solution, 8.00 N	100 mL MDB**	282-32
Sulfuric Acid, 5.25 N	100 mL MDB	2449-32
Water, deionized	4 L	272-56
Select one or more based on sample concentration:		
EDTA Titration Cartridge, 0.0800 M	each	14364-01
EDTA Titration Cartridge, 0.1428 M	each	14960-01
EDTA Titration Cartridge, 0.714 M	each	14959-01
EDTA Titration Cartridge, 0.800 M	each	14399-01

REQUIRED APPARATUS

Digital Titrator	each	16900-01
Flask, Erlenmeyer, 250 mL	each	505-46
Select one or more based on sample concentration:		
Cylinder, graduated, 10 mL	each	508-38
Cylinder, graduated, 25 mL	each	508-40
Cylinder, graduated, 50 mL	each	508-41
Cylinder, graduated, 100 mL	each	508-42

OPTIONAL REAGENTS

CDTA Magnesium Salt Powder Pillows	100/pkg	14080-99
CDTA Titration Cartridge, 0.08 M	each	14402-01
CDTA Titration Cartridge, 0.8 M	each	14403-01
Hardness 2 Test Solution	100 mL MDB	425-32
Hardness Standard Solution, Voluette® Ampules, 10,000 mg/L as $CaCO_3$, 10 mL	16/pkg	2187-10
Hydroxylamine Hydrochloride	113 g	246-14
Nitric Acid, 70%	500 mL	152-49

* Other reagents and apparatus are listed with the specific procedure.

** Marked Dropper Bottle (MDB). Contact Hach for larger sizes.

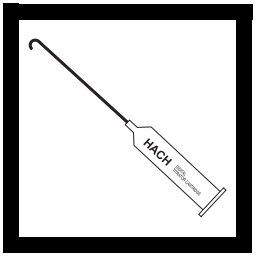
OPTIONAL APPARATUS

Description	Unit	Cat. No.
Bottle, wash, poly, 500 mL	each	620-11
Clamp, 2-prong, extension, 38 mm	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473 mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook	5/pkg	41578-00
pH Paper, 1.0 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, volumetric, Class A, 10 mL	each	14515-38
Pipet, volumetric, Class A, 20 mL	each	14515-20
Pipet, volumetric, Class A, 25 mL	each	14515-40
Pipet, volumetric, Class A, 50 mL	each	14515-41
Pipet, volumetric, Class A, 100 mL	each	14515-42
Pipet Filler, safety bulb	each	14651-00
*sens**ion**™1* Basic Portable pH Meter with electrode	each	51700-10
Spoon, measuring, 0.1 gram	each	511-00
Spoon, measuring, 0.5 gram	each	907-00
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10
Voluette® Ampule Breaker Kit	each	21968-00

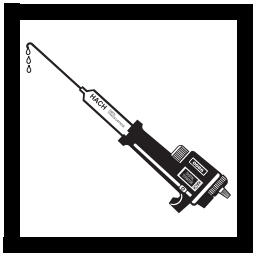
HYPOCHLORITE (Bleach) (50 to 150 g/L [5 to 15%] as Cl2)

Iodometric Method*

Scope and Application: For testing concentrated liquid bleach (sodium hypochlorite, soda bleach) used as a disinfectant in drinking water or wastewater treatment.



1. Insert a clean delivery tube into the 2.26 N Thiosulfate Titrant Solution cartridge. Attach the cartridge to the titrator body.

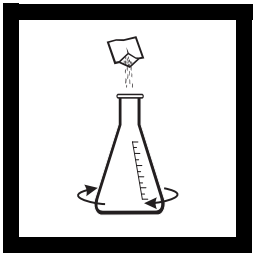


2. Flush the delivery tube by turning the deliver knob to eject a few drops of titrant. Reset the counter to zero and wipe off the tip.

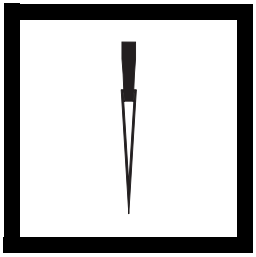
3. Fill a 125-mL Erlenmeyer flask to about the 75-mL mark with deionized or tap water.

Note: *The level of residual chlorine found in tap water will not interfere in the test.*

4. Add the contents of one Potassium Iodide Powder Pillow to the flask and swirl to mix.



5. Add the contents of one Acid Reagent Powder Pillow to the flask and swirl to mix.



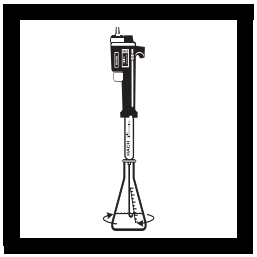
6. Attach a clean tip to the TenSette® Pipet.

7. Use the pipet to dispense 0.2 mL of bleach sample below the solution level in the flask.

8. Swirl to mix. The solution will turn dark brown.

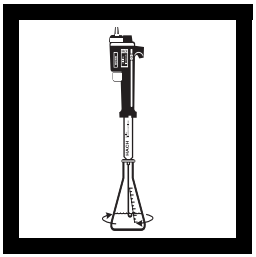
Note: *Proceed immediately with Step 9.*

* Adapted from ASTM method D2022.



9. Place the delivery tube tip into the solution and swirl the flask while titrating with the thiosulfate titrant until the solution is pale yellow.

10. Add one dropper of Starch Indicator Solution to the flask and swirl to mix. A dark blue or green color will develop.



11. Continue the titration until the solution becomes colorless. Record the number of digits required.

g/L Chlorine as Cl_2 = Digits at End Point x 0.5

12. Calculate the g/L chlorine:

g/L chlorine = Digits Required x 0.5

Note: *Divide the g/L chlorine by 10 to obtain the % (by volume) chlorine ("trade percent").*

Sample Collection, Preservation and Storage

Soda bleach solutions are relatively unstable. Avoid exposing the sample to heat or light. Collect samples in glass bottles and store in a cool, dark place until analyzed. Analyze as soon as practical.

Accuracy Check

Standard Solution Method

Hach strongly recommends that, for optimum test results, reagent accuracy be checked with each new lot of reagents. The strength of the Thiosulfate Standard Solution can be checked using Potassium Iodide-Iodate Standard Solution:

1. Use a Class A pipet to transfer 50.00 mL of 0.0125 N Potassium Iodide-Iodate Standard Solution to a clean 125-mL Erlenmeyer flask.

2. Add the contents of one Potassium Iodide Powder Pillow to the flask and swirl to mix.

3. Add the contents of three Acid Reagent Powder Pillows to the flask and swirl to mix. Swirl until all powder is dissolved.

4. Continue the titration starting at *step 9* of the procedure. It should take 217–227 digits of 2.26 N Thiosulfate Standard Solution to reach the end point.

Interferences

The iodometric method is relatively free of interferences. The test will determine chlorite ion (ClO_2^-) in addition to the hypochlorite ion (ClO^-). However, the amount of chlorite in commercial bleach is insignificant (typically less than 0.2%).

A large excess of caustic in the bleach sample may lead to low results. After adding the Acid Reagent Powder Pillow (*step 5*), check the pH of the solution with pH paper. The pH should be less than 3. If not, add additional Acid Reagent, one pillow at a time, until the pH drops below 3.

For most accurate results, the temperature of the dilution water (*step 3*) should be less than 20 °C (68 °F).

Precision

In a single laboratory, using a commercial bleach sample of 91.2 g/L (9.12%) Cl_2, a single operator obtained a standard deviation of ± 1.5 g/L (± 0.15%) Cl_2.

Summary of Method

Under acidic conditions, hypochlorite reacts with iodide to produce an equivalent amount of triiodide (I_3^-). The released I_3^- is titrated with standard thiosulfate solution to a colorless end point. The number of digits of thiosulfate required is proportional to the hypochlorite concentration in the original bleach sample.

REQUIRED REAGENTS

HR Hypochlorite (Bleach) Reagent Set (about 100 tests) 26870-00
Includes: (1) 349-32, (1) 1042-99, (1) 20599-96, (1) 26869-01

Description	Unit	Cat. No.
Acid Reagent Powder Pillows	100/pkg	1042-99
Potassium Iodide Powder Pillows	50/pkg	20599-96
Sodium Thiosulfate Standard Titrant Solution, 2.26 N	each	26869-01
Starch Indicator Solution	100 mL MDB*	349-32

REQUIRED APPARATUS

Clippers, large	each	968-00
Delivery Tubes, 180°	5/pkg	17205-00
Digital Titrator Assembly	each	16900-02
Flask, Erlenmeyer, 125-mL	each	505-43
Pipet, TenSette®, 0.1–1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette® Pipet	50/pkg	21856-96

OPTIONAL REAGENTS

Potassium Iodide-Iodate Standard Solution, 0.0125 N	1 L	14001-53

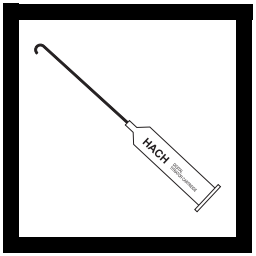
OPTIONAL APPARATUS

pH Paper, 1–11	5/pkg	391-33
Pipet, volumetric, Class A, 50.00 mL	each	14515-41

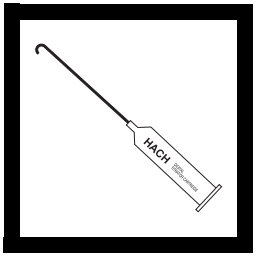
* Marked Dropping Bottle

IRON (10 to 1000 mg/L as Fe)

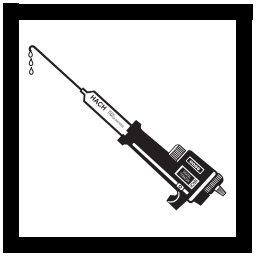
Using the TitraVer® Titration Cartridge



1. Select a sample volume and a TitraVer Titration Cartridge corresponding to the expected iron (Fe) concentration from *Table 1*.



2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step*, for assembly instructions, if necessary.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Use a graduated cylinder to measure the sample volume from *Table 1*. Transfer the sample into a clean 125-mL Erlenmeyer flask. Dilute to about the 50-mL mark with deionized water, if necessary.

Table 1

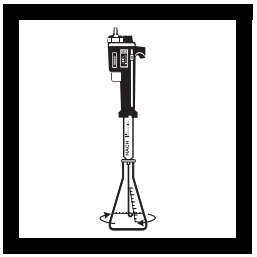
Range (mg/L as Fe)	Sample Volume (mL)	Titration Cartridge (M TitraVer)	Catalog Number	Digit Multiplier
10-40	50	0.0716	20817-01	0.1
25-100	20	0.0716	20817-01	0.25
100-400	50	0.716	20818-01	1.0
250-1000	20	0.716	20818-01	2.5

5. Add the contents of one Citrate Buffer Powder Pillow and swirl to mix.

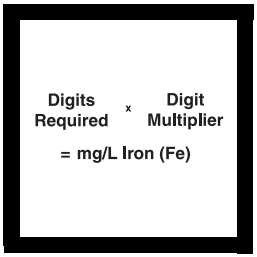
6. Add the contents of one Sodium Periodate Powder Pillow and swirl to mix.

Note: *A yellow color indicates the presence of iron.*

7. Add the contents of one Sulfosalicylic Acid Powder Pillow and swirl to mix. A red color will develop if iron is present.



8. Place the delivery tube tip into the solution and swirl the flask while titrating the sample until the color changes from red to the original yellow. Record the number of digits required.



9. Calculate:

Digits Required x Digit Multiplier = mg/L Iron (Fe)

Accuracy Check

This accuracy check should be performed when interferences are suspected or to verify analytical technique.

1. Use a TenSette® Pipet to add 0.5 mL of 1000 mg/L as Fe standard to the sample in *step 7*. Resume titration back to the same end point. Record the number of additional digits required.

2. Repeat, using two more additions of 0.5 mL. Titrate to the end point after each addition.

3. Each 0.5-mL addition of standard should require 10 additional digits of 0.716 M titrant or 100 digits of 0.0716 M titrant. If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

Summary of Method

Iron (Fe^{2+}) is oxidized by sodium periodate to the ferric ion (Fe^{3+}). When sulfosalicylic acid is present, the ferric ion forms a red complex, coloring the solution. The red complex is destroyed by titration with EDTA. Citric acid is used to buffer the solution and to stabilize the ferric ion in solution.

REQUIRED REAGENTS

(varies with sample characteristics)

Iron Reagent Sets (about 100 tests)

10-100 mg/L includes: (1) 984-99, (1) 20815-99, (1) 20816-69, (1) 20817-01 24492-00

100-1000 mg/L includes: (1) 984-99, (1) 20815-99, (1) 20816-69, (1) 20818-01 24493-00

Description	Unit	Cat. No.
Citrate Buffer Powder Pillows	100/pkg	20815-99
Sodium Periodate Powder Pillows	100/pkg	984-99
Sulfosalicylic Acid Powder Pillows	100/pkg	20816-69
TitraVer® Standard Solution Titration Cartridge, 0.0716 M	each	20817-01
TitraVer® Standard Solution Titration Cartridge, 0.716 M	each	20818-01
Water, deionized	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening pillows	each	968-00
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 125 mL	each	505-43
Select one or more based on sample concentration:		
Cylinder, graduated, 25 mL	each	508-40
Cylinder, graduated, 50 mL	each	508-41

OPTIONAL REAGENTS

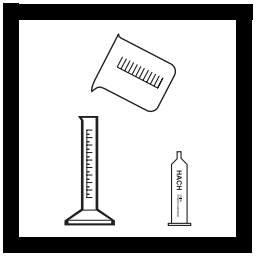
Iron Standard Solution, 1000 mg/L as Fe	100 mL	2271-42

OPTIONAL APPARATUS

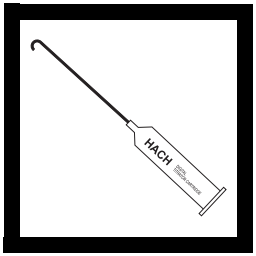
Bottle, wash, poly, 500 mL	each	620-11
Clamp, 2-prong, extension, 38 mm	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473 mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook	5/pkg	41578-00
Pipet, TenSette®, 0.1 to 1.0 mL	each	19700-01
Pipet Tips for 19700-01 TenSette® Pipet	50/pkg	21856-96
Pipet, volumetric, Class A, 25.0 mL	each	14515-40
Pipet, volumetric, Class A, 50.0 mL	each	14515-41
Pipet Filler, safety bulb	each	14651-00
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

NITRITE (100 to 2500 mg/L as $NaNO_2$)

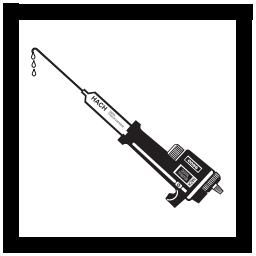
Using Ceric Standard Solution



1. Select the sample volume from *Table 1* which corresponds to the expected sample sodium nitrite concentration (as $NaNO_2$).



2. Insert a clean delivery tube into the Ceric Standard Solution Titration Cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step,* for assembly instructions, if necessary.



3. Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob until a few drops of titrant are expelled. Reset the counter to zero and wipe the tip.

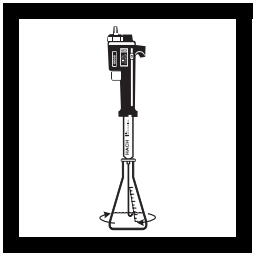
Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample to a clean 125-mL Erlenmeyer flask. Add deionized water to about the 75-mL mark, if necessary.

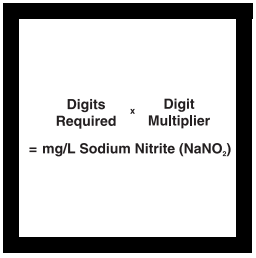
Note: *A pipet is recommended for sample volumes less than 10 mL.*

5. Add five drops of 5.25 N Sulfuric Acid Standard Solution to the flask. Swirl to mix.

6. Add one drop of Ferroin Indicator Solution to the flask. Swirl to mix.



7. Place the delivery tip into the solution. While titrating with Ceric Standard Solution, swirl the flask until the solution color changes from orange to pale blue. Record the number of digits required.



8. Calculate:

Digits Required x Digit Multiplier = mg/L Sodium Nitrite ($NaNO_2$)

Note: *See Standardization of Ceric Solution to verify the normality.*

Table 1

Expected Sample Concentration (as $NaNO_2$)	Sample Volume (mL)	Digit Multiplier
100-400	25	0.86
400-800	10	2.15
800-1500	5	4.31
1500-2500	2	10.78

Standardization of Ceric Solution

The normality of the Ceric Solution will sometimes decrease over time. Before use, verify the normality with the following procedure. This standardization should be done monthly.

1. Use a graduated cylinder or pipet to measure 50 mL of deionized water into a 125-mL Erlenmeyer flask.
2. Add 5 mL of 19.2 N Sulfuric Acid Standard Solution. Swirl to mix.
3. Insert a clean delivery tube into a Ceric Standard Titration Cartridge.
4. Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob until a few drops of titrant are expelled. Reset the counter to zero and wipe the tip.
5. Place the delivery tube tip into the solution. While swirling the flask, add 200 digits of Ceric Standard.
6. Insert a clean delivery tube into a 0.200 N Sodium Thiosulfate Titration Cartridge.
7. Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob until a few drops of titrant are expelled. Reset the counter to zero and wipe the tip.
8. Place the delivery tube tip into the solution. While swirling the flask, titrate with the sodium thiosulfate from an intense yellow color to a faint yellow color. Record the number of

digits required. This step should require about 400-450 digits of titrant.

9. Add one drop of Ferroin Indicator Solution. Swirl to mix. The solution will turn a faint blue.

10. Continue titrating the Ceric Standard Solution (using the 0.20 Sodium Thiosulfate Titration Cartridge) from a faint blue to orange color. Record the number of digits required.

11. Calculate the correction factor:

$$\text{Correction Factor} = \frac{\text{Digits Required}}{500}$$

12. Multiply the mg/L sodium nitrite from *step 8* of the nitrite titration procedure by the correction factor to obtain the correct sodium nitrite concentration.

Sampling and Storage

Collect samples in clean plastic or glass bottles. Prompt analysis is recommended.

If prompt analysis is impossible, store samples for 24 to 48 hours at 4 °C (39 °F) or lower. Warm to room temperature before analysis. Do not use acid preservatives.

Accuracy Check

Dissolve 1.000 gram of fresh sodium nitrite in 100 mL of deionized water. Dilute to 1000 mL with deionized water to prepare a 1000 mg/L sodium nitrite standard solution. Use a 5.0 sample of the standard solution and start with *step 4* of the titration procedure. The analysis should yield 1000 mg/L for *step 8* of the titration procedure.

Summary of Method

Sodium nitrite is titrated with tetravalent cerium ion, a strong oxidant, in the presence of ferroin indicator. After the cerium oxidizes the nitrite, it oxidizes the indicator, causing a color change from orange to pale blue. The concentration of sodium nitrite is proportional to the amount of titrant used.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Ceric Standard Solution Titration Cartridge, 0.5 N	each	22707-01
Ferroin Indicator Solution	29 mL DB	1812-33
Sulfuric Acid Standard Solution, 5.25 N	100 mL MDB	2449-32

REQUIRED APPARATUS

Digital Titrator	each	16900-01
Flask, Erlenmeyer, 125 mL	each	505-43
Select one or more based on sample volume:		
Cylinder, graduated, 100 mL, poly	each	1081-42
Pipet, serological, 10 mL	25/pkg	20931-28

REQUIRED APPARATUS (Using Titrastir® Stir Plate Modification)

Delivery Tubes, 90° with hook	5/pkg	41578-00
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 125 mL	each	505-43
Stir Bar, octagonal, Teflon-coated, 50.8 x 7.9 mm	each	20953-55
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

OPTIONAL REAGENTS

Sodium Thiosulfate Titration Cartridge, 0.200 N	each	22675-01
Sodium Nitrite, ACS	454 g	2452-01
Sulfuric Acid Standard Solution, 19.2 N	100 mL	2038-32
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

Balance, electronic, analytical	each	22310-00
Flask, volumetric, Class A, 50 mL	each	14547-41

OXYGEN, DISSOLVED (1 to greater than 10 mg/L as DO)

Azide Modification of Winkler Method

Using a 300-mL BOD Bottle — Method 8215

1. Collect a water sample in a clean 300-mL BOD Bottle.

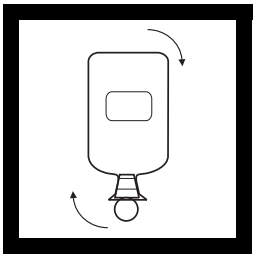
Note: *Allow the sample to overflow the bottle for 2-3 minutes to ensure air bubbles are not trapped.*

Note: *If samples cannot be analyzed immediately, see Sampling and Storage on page 152.*

2. Add the contents of one Manganous Sulfate Powder Pillow and one Alkaline Iodide-Azide Reagent Powder Pillow.

3. Immediately insert the stopper so air is not trapped in the bottle. Invert several times to mix.

Note: *A flocculent precipitate will form. It will be orange-brown if oxygen is present or white if oxygen is absent. The floc settles slowly in salt water and normally requires 5 additional minutes before proceeding to step 5.*



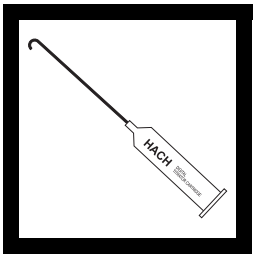
4. Wait until the floc in the solution has settled. Again invert the bottle several times and wait until the floc has settled.

Note: *Waiting until floc has settled twice assures complete reaction of the sample and reagents.*

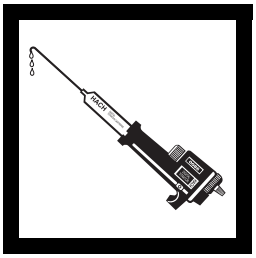
5. Remove the stopper and add the contents of one Sulfamic Acid Powder Pillow. Replace the stopper without trapping air in the bottle and invert several times.

Note: *The floc will dissolve and leave a yellow color if oxygen is present.*

6. Select a sample volume and Sodium Thiosulfate Titration Cartridge corresponding to the expected dissolved oxygen (D.O.) concentration from *Table 1*.



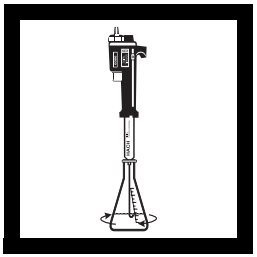
7. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step* for assembly instructions, if necessary.



8. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

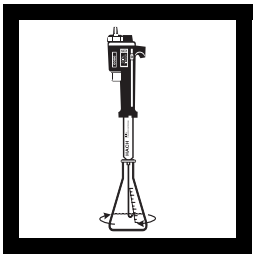
9. Use a graduated cylinder to measure the sample volume from *Table 1*. Transfer the sample into a 250-mL Erlenmeyer flask.



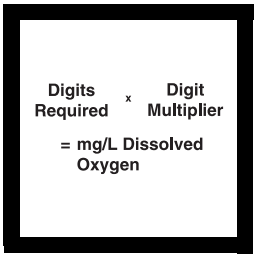
10. Place the delivery tube tip into the solution and swirl the flask while titrating with sodium thiosulfate to a pale yellow color.

11. Add two 1-mL droppers of Starch Indicator Solution and swirl to mix.

Note: *A dark blue color will develop.*



12. Continue the titration to a colorless end point. Record the number of digits required.



13. Calculate:

Digits Required x Digit Multiplier = mg/L Dissolved Oxygen

Table 1

Range (mg/L D.O.)	Sample Volume (mL)	Titration Cartridge (N $Na_2S_2O_3$)	Catalog Number	Digit Multiplier
1-5	200	0.200	22675-01	0.01
2-10	100	0.200	22675-01	0.02
>10	200	2.000	14401-01	0.1

Using a 60-mL BOD Bottle **Method 8332**

1. Collect a water sample in a clean 60-mL glass-stoppered BOD Bottle.

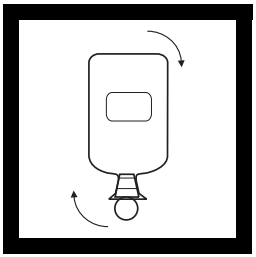
Note: *Allow the sample to overflow the bottle for 2-3 minutes to ensure air bubbles are not trapped.*

Note: *If samples cannot be analyzed immediately, see Sampling and Storage on page 152.*

2. Add the contents of one Dissolved Oxygen 1 Reagent Powder Pillow and one Dissolved Oxygen 2 Reagent Powder Pillow.

3. Immediately insert the stopper so air is not trapped in the bottle. Invert several times to mix.

Note: *A flocculent precipitate will form. It will be orange-brown if oxygen is present or white if oxygen is absent. The floc settles slowly in salt water and normally requires 5 additional minutes before proceeding to step 5.*



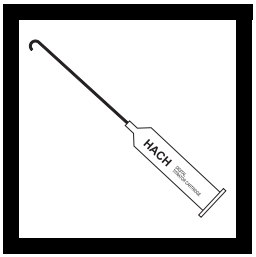
4. Wait until the floc in the solution has settled and the top half of the solution is clear. Again invert the bottle several times and wait until the floc has settled.

Note: *Results are not affected if the floc does not settle or if some of the reagent powder does not dissolve.*

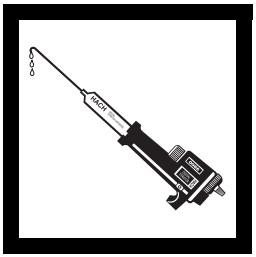
5. Remove the stopper and add the contents of one Dissolved Oxygen 3 Powder Pillow. Replace the stopper without trapping air in the bottle and invert several times to mix.

Note: *The floc will dissolve and leave a yellow color if oxygen is present.*

6. Accurately measure 20 mL of the prepared sample and transfer it to a 125-mL Erlenmeyer flask.

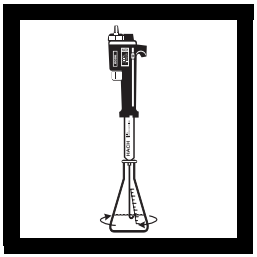


7. Attach a clean straight-stem delivery tube to a 0.2000 N Sodium Thiosulfate Titration Cartridge. Twist the cartridge onto the titrator body. See *General Description, Step-by-Step* for assembly instructions, if necessary

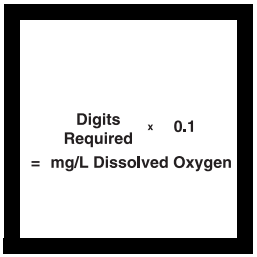


8. Flush the delivery tube by turning the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir Stir Plate. See General Description, Step 3 in Step-by-Step.*



9. Titrate the prepared solution with 0.2000 N Sodium Thiosulfate until the sample changes from yellow to colorless. Record the number of digits.



10. Calculate:

Digits required x 0.1 = mg/L Dissolved Oxygen

Sampling and Storage

Sampling and sample handling are important in obtaining meaningful results. The dissolved oxygen content of the sample changes with depth, turbulence, temperature, sludge deposits, light, microbial action, mixing, travel time, and other factors. A single dissolved oxygen test rarely reflects the over-all condition of a body of water. Several samples taken at different times, locations and depths are recommended for most reliable results.

Collect samples in clean BOD Bottles (see *step 1*). If storage is necessary, run steps 1-5 of the procedure and store in the dark at 10-20 ° C. Seal the bottle with water by pouring a small volume of water into the flared lip area of a stopper bottle. Snap a BOD Bottle Cap over the flared lip. Samples preserved like this can be held 4-8 hours. Begin with *step 6* when analyzing.

Accuracy Check

Check the strength of the Sodium Thiosulfate Solution by using an Iodate-Iodide Standard Solution which is equivalent to 10 mg/L dissolved oxygen. For the 300-mL procedure, begin at *step 5* adding the Sulfamic Acid Powder Pillow. For the 60-mg/L procedure, begin the analysis at *step 5* adding the Dissolved Oxygen 3 Powder Pillow. The titration should take 10 mL. If

more than 10.5 mL is required to reach the end point, replace the Sodium Thiosulfate Solution.

Interferences

Nitrite interference is eliminated by the azide in the reagents. Other reducing or oxidizing substances may interfere. If these are present, use an alternate method, such as the High Range Dissolved Oxygen Method (colorimetric) in this manual, or a dissolved oxygen electrode.

Summary of Method

Samples are treated with manganous sulfate and alkaline iodide-azide reagent to form an orange-brown precipitate. Upon acidification of the sample, this floc reacts with iodide to produce free iodine as triiodide in proportion to the oxygen concentration. The iodine is titrated with sodium thiosulfate to a colorless end point.

REQUIRED REAGENTS FOR 300-ML BOD BOTTLE

(Varies with sample characteristics)

Dissolved Oxygen Reagent Set (about 50 tests) 22722-00
Includes: (1) 349-32, (2) 1071-68, (2) 1072-68, (1) 22675-01, (2) 20762-68

Description	Unit	Cat. No.
Alkaline Iodide-Azide Powder Pillows	25/pkg	1072-68
Manganous Sulfate Powder Pillows	25/pkg	1071-68
Sodium Thiosulfate Titration Cartridge, 0.2000 N	each	22675-01
Sodium Thiosulfate Titration Cartridge. 2.00 N	each	14401-01
Starch Indicator Solution	100 mL MDB*	349-32
Sulfamic Acid Powder Pillows	25/pkg	20762-68

REQUIRED APPARATUS FOR 300-ML BOD BOTTLE

Bottle, with stopper, BOD, 300-mL	each	621-00
Clippers, for opening pillows	each	968-00
Cylinder, graduated, 250-mL	each	508-46
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 250-mL	each	505-46

* Contact Hach for larger sizes.

REQUIRED REAGENTS FOR 60-ML BOD BOTTLE

Description	Unit	Cat. No.
Dissolved Oxygen 1 Reagent Powder Pillows	100/pkg	981-99
Dissolved Oxygen 2 Reagent Powder Pillows	100/pkg	982-99
Dissolved Oxygen 3 Reagent Powder Pillows	25/pkg	987-68
Sodium Thiosulfate Titration Cartridge, 0.2000 N	each	22675-01

REQUIRED APPARATUS FOR 60-ML BOD BOTTLE

Bottle, with stopper, BOD, 60-mL	each	1909-02
Clippers, for opening pillows	each	968-00
Cylinder, graduated, 50-mL	each	1081-41
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 125 mL	each	505-43

OPTIONAL REAGENTS

Iodate-Iodide Standard Solution, 10 mg/L as DO	500 mL*	401-49

OPTIONAL APPARATUS

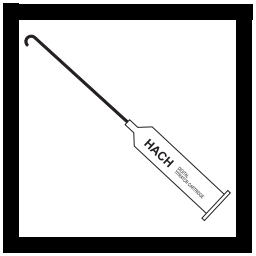
Cap, BOD Bottle, plastic	6/pkg	2419-06
Clamp Holder	each	326-00
Clamp, 2-prong, extension, 38 mm	each	21145-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook	5/pkg	41578-00
Sewage Sampler, Lab-Line	each	427-00
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

Procedures and kits for portable dissolved oxygen measurements using this method are available from Hach.

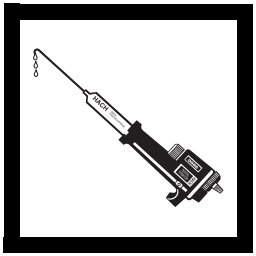
* Contact Hach for larger sizes.

SALINITY (0 to 100 ppt* as Salinity)

Using Mercuric Nitrate

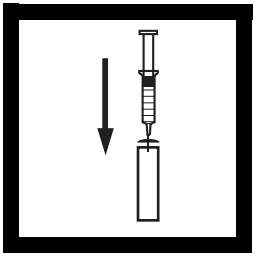


1. Insert a clean, straight-stem delivery tube to a Mercuric Nitrate Titration Cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step* for assembly instructions, if necessary.

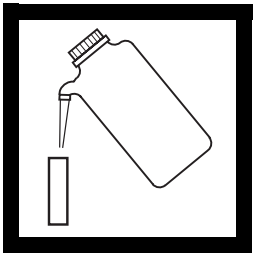


2. Flush out the delivery tube by turning the knob until titrant begins flowing from the end of the tube. Wipe the tip and reset the counter to zero.

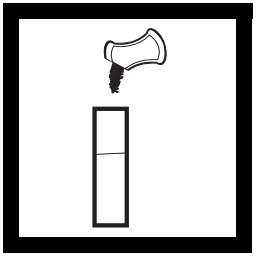
Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*



3. Using the 3-mL (3-cc) syringe, collect a 2.0-mL water sample. Add to the vial provided.

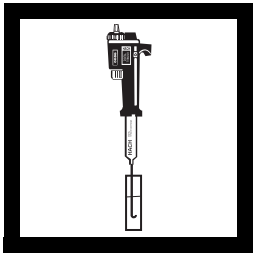


4. Fill the vial to the 10-mL mark with deionized water.



5. Add the contents of one Diphenylcarbazone Reagent Powder Pillow to the vial and mix.

Note: *Results will not be affected if a small portion of the diphenylcarbazone reagent powder does not dissolve.*



6. Titrate the sample with mercuric nitrate until the color changes from yellow to light pink.

Record the number of digits.

7. Determine the salinity of the water sample in parts per thousand (ppt) by multiplying the reading by 0.1.

Note: *Results may be expressed as mg/L Cl^- by multiplying the ppt salinity by 569. Results may be expressed as mg/L NaCl by multiplying the ppt salinity by 940.*

* ppt = parts per thousand

Summary of Method

The mercuric nitrate method of chloride analysis has become popular due to the sharp yellow to pinkish-purple end point of diphenylcarbazone. A single, stable powder has been developed, combining the color indicator with an appropriate buffer to establish the correct pH.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Diphenylcarbazone Reagent Powder Pillows	100/pkg	967-99
Mercuric Nitrate Titration Cartridge, 2.570 N	each	23937-01

REQUIRED APPARATUS

Vial, 2, 5, 10, 15, 20, 25-mL marks	each	2193-00
Syringe, 3 cc, Luer lock tip	each	43213-00
Demineralizer Assembly, 473 mL	each	21846-00

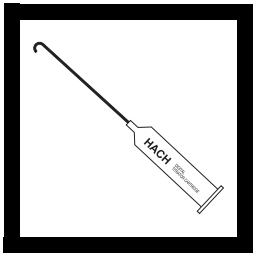
OPTIONAL APPARATUS

TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

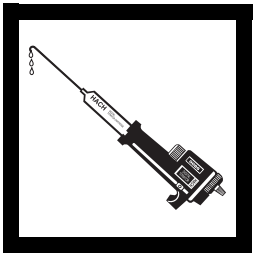
SULFITE (4 to greater than 400 mg/L as SO_3^{2-})

Using Iodate-Iodide

1. Select a sample volume corresponding to the expected sulfite (SO_3^{2-}) concentration from *Table 1*.



2. Insert a clean delivery tube into the Iodate-Iodide Titration Cartridge (KIO_3-KI). Attach the cartridge to the titrator body. See *General Description, Step-by-Step*, for assembly instructions, if necessary.



3. Turn the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample into a clean 125-mL Erlenmeyer flask. Dilute to about the 50-mL mark with deionized water.

Note: *Avoid unnecessary agitation throughout the procedure.*

Note: *See Sampling and Storage on page 158.*

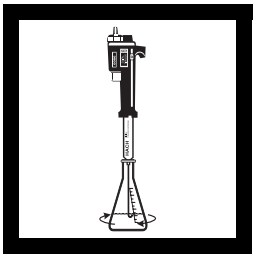
Table 1

Range (mg/L as SO_3^{2-})	Sample Volume (mL)	Titration Cartridge (N KIO_3-KI)	Catalog Number	Digit Multiplier
Up to 160	50	0.3998	14961-01	0.4
100-400	20	0.3998	14961-01	1.0
200-800	10	0.3998	14961-01	2.0
>400	5	0.3998	14961-01	4.0

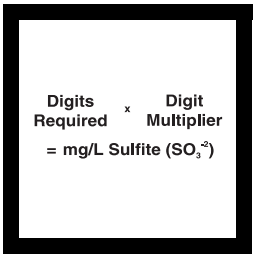
5. Add the contents of one Dissolved Oxygen 3 Reagent Powder Pillow and swirl gently to mix.

Note: *0.5 mL of 19.2 N Sulfuric Acid Standard Solution may be substituted for the powder pillow.*

6. Add one dropperful of Starch Indicator Solution and swirl to mix.



7. Place the delivery tube tip into the solution and swirl the flask while titrating with the iodate-iodide to a permanent blue end point. Record the number of digits required.



8. Calculate:

Digits Required x Digit Multiplier = mg/L Sulfite (SO_3^{2-})

Note: *To obtain the concentration of other sulfite forms, multiply the mg/L SO_3^{2-} determined in step 8 by the appropriate multiplier from Table 2.*

Table 2

Form	Multiplier
Bisulfite, Hydrogen Sulfite (HSO_3^-)	1.01
Sodium Bisulfite, Sodium Hydrogen Sulfite ($NaHSO_3$)	1.30
Sodium Metabisulfite, Sodium Pyrosulfite ($Na_2S_2O_5$)	1.19
Sodium Sulfite (Na_2SO_3)	1.58

Sampling and Storage

Samples must be analyzed immediately. Cool hot samples to 50 °C or lower.

Accuracy Check

Standard Additions Method

This accuracy check should be performed when interferences are suspected or to verify analytical technique.

1. Snap the neck off a Sulfite Standard Solution Voluette® Ampule, 5,000 mg/L SO_3^{2-}.

2. Use a TenSette® Pipet to add 0.1 mL of standard to the sample titrated in *step 7*. Resume titration back to the same end point. Record the number of digits required.

3. Repeat, using additions of 0.2 and 0.3 mL, titrating to the end point after each.

4. Each 0.1 mL addition of standard should require 25 additional digits of titrant. If these uniform increases do not occur, refer to *Appendix A, Accuracy Check and Standard Additions*.

A standard solution equivalent to 40 mg/L sulfite can be prepared by diluting 10.0 mL of 0.025 N Sodium Thiosulfate Titrant to 250 mL in a volumetric flask. Titrate a 50 mL sample, using the above procedure.

Interferences

Sulfide, organic matter and other oxidizable substances will cause positive error in the titration. Nitrite will react with sulfite to cause low results. Some metals, especially copper, catalyze the oxidation of sulfite to sulfate. Addition of one Dissolved Oxygen 3 Powder Pillow per liter of sample immediately upon sampling will help eliminate the effects of nitrite and copper.

Summary of Method

Sulfite ion is titrated with potassium iodate-iodide standard solution under acidic conditions to a blue starch end point. The volume of titrant used is proportional to the sulfite concentration.

REQUIRED REAGENTS

Sulfite Reagent Set (about 100 tests) 22723-00
Includes: (1) 349-32, (1) 987-99, (1) 14961-01

Description	Unit	Cat. No.
Dissolved Oxygen 3 Reagent Powder Pillows	100/pkg	987-99
Iodate-Iodide Titration Cartridge, 0.3998 N	each	14961-01
Starch Indicator Solution	100 mL MDB*	349-32
Water, deionized	4 L	272-56

* Contact Hach for larger sizes.

REQUIRED APPARATUS

Description	Unit	Cat. No.
Clippers, for opening pillows	each	968-00
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 125 mL	each	505-43
Select one or more based on sample concentration:		
Cylinder, graduated, 10 mL	each	508-38
Cylinder, graduated, 25 mL	each	508-40
Cylinder, graduated, 50 mL	each	508-41

OPTIONAL REAGENTS

Sodium Thiosulfate Standard Solution, 0.025 N	1000 mL	24093-53
Sulfite Standard Solution, Voluette® Ampules, 5,000 mg/L SO_3 10 mL	16/pkg	14267-10
Sulfuric Acid Standard Solution, 19.2 N	100 mL MDB	2038-32

OPTIONAL APPARATUS

Bottle, wash poly, 500 mL	each	620-11
Clamp, 2-prong, extension, 38 mm	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473 mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook	5/pkg	41578-00
Flask, volumetric, Class B, 250 mL	each	547-46
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, 5 mL	each	14515-37
Pipet, volumetric, Class A, 10 mL	each	14515-38
Pipet, volumetric, Class A, 20 mL	each	14515-20
Pipet, volumetric, Class A, 50 mL	each	14515-41
Pipet Filler, safety bulb	each	14651-00
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10
Voluette® Ampule Breaker Kit	each	21968-00

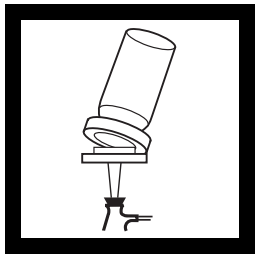
TURBIDITY STANDARDS

Preparing Turbidity-Free Water

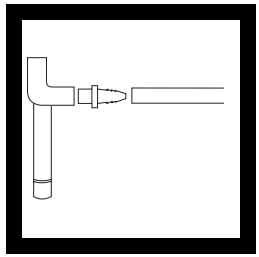
Phase 1: Filtration Assembly

1. Attach the filter funnel stem to a 1000-mL filtering flask.

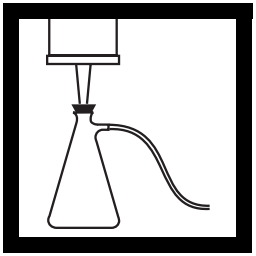
2. Using plastic tweezers, position a 0.45 micron membrane filter on top of the funnel stem.



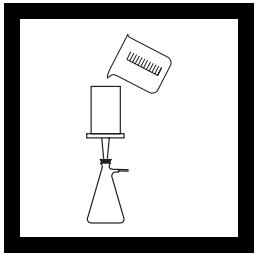
3. Cover the stem with the filter housing.



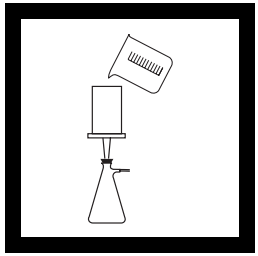
4. Attach a vacuum hose to the water aspirator and turn on water.



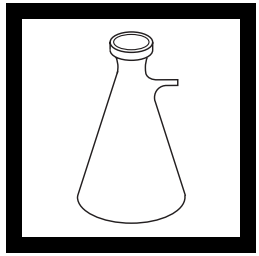
5. Attach the vacuum hose to the sidearm.



6. Pour a total of about 800 mL, in three portions, of deionized water through the filter funnel and wait until it passes through the filter. Discard this rinse water.

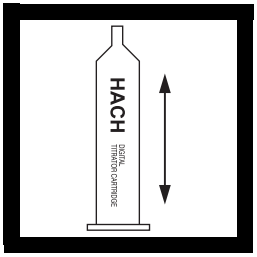


7. Again add 800 mL of deionized water through the filter funnel and collect in a filter flask. Remove vacuum hose from sidearm and turn off water.

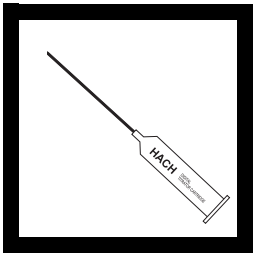


8. Use this filtered deionized water for all formazin dilutions.

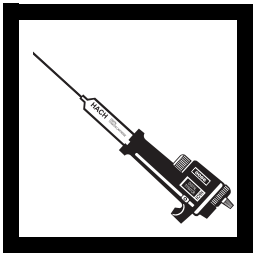
Phase 2: Preparing Standards using a Formazin Cartridge



1. Shake the Formazin Cartridge vigorously for one minute to mix the formazin suspension.

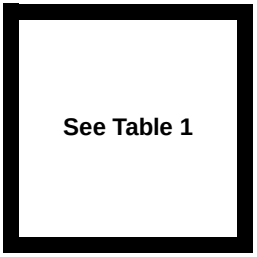


2. Attach a clean delivery tube to the 4000 NTU Formazin Cartridge. Cut the hooked end off the delivery tube with clippers. Attach the cartridge to the titrator body. See *General Description, Step-by-Step*, for assembly instructions, if necessary.



3. Flush the delivery tube by turning the delivery knob until a few drops of formazin are ejected from the tube. Zero the counter and wipe the tip.

Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*



4. Select the standard concentration from the list below. Dispense the formazin into a clean Class A volumetric flask. Dilute with turbidity-free water to the mark and mix well. Refer to the table below for the standard(s) you require.

Table 1

Formazin Standard Concentration (NTU)	Number of Digits Required	Flask Size
100	1000	50-mL
40	400	50-mL
10	100	50-mL
4	80	100-mL

Prepararation of a 1 NTU Formazin Standard

There will be a residual amount of turbidity in even the purest water used to make formazin dilutions. At the 1 NTU level this can affect the value of the formazin standard significantly, causing a positive error. The following procedure corrects for the turbidity of the dilution water when making a 1 NTU formazin standard in a 500-mL, Class A, volumetric flask.

1. Standardize the turbidimeter with a secondary standard on a range appropriate for the measurement of the dilution water, usually the 0-1 NTU range.

2. Measure and record the turbidity of the dilution water to be used in making the 1 NTU formazin standard.

3. Calculate the number of digits necessary to dispense the proper amount of formazin into a 500-mL, Class A, volumetric flask for a 1 NTU formazin standard:

   $$\text{DIGITS} = 100(1 - T_W)$$

   Where:

   T_W is the turbidity of the dilution water

4. Carefully dispense the calculated number of digits into a 500-mL volumetric flask. Dilute with dilution water to the 500-mL mark and mix well.

Preparation of any Formazin Standard

The following formula may be used to determine the correct number of digits necessary to dispense formazin for a standard of any value.

$$\text{DIGITS} = (0.2)(V)(T_D - T_W)$$

Where:

T_D = desired turbidity of the formazin standard

T_W = turbidity of the dilution water (this term may be dropped if it is 1% or less of the TD value)

V = volume of the flask in mL

Example 1:
One liter of a 0.5 NTU formazin standard is required. It is found that the dilution water has a turbidity of 0.05 NTU. Because the dilution water turbidity is 10% of the desired standard, the dilution water correction must be made.

The number of digits of formazin is equal to:

DIGITS = (0.2) (1000.0) (0.5-0.05) = 90

Thus, 90 digits of formazin dispensed in a 1000 mL, Class A, volumetric flask and diluted to volume with 0.05 NTU water will give a 0.5 NTU formazin standard. The size of the volumetric flask should be chosen so that the number of digits calculated is approximately 100 or more.

Example 2:
50 mL of a 50 NTU formazin standard is required. It is found that the dilution water is 0.1 NTU. Because the dilution water is only 0.2% of the desired standard, the dilution water correction can be ignored.

The number of digits necessary to dispense the formazin is:

DIGITS = (0.2) (50.0) (50) = 500

500 digits of formazin diluted with 50.0 mL of dilution water will give a 50 NTU formazin standard.

Interferences

Because dirty or scratched glassware, air bubbles and color in a sample will interfere with turbidity measurements, sample cells must be scratch-free and samples should be colorless without air bubbles.

Summary of Method

The measurement of turbidity is based on the scattering of light by the suspended particles (clay, sand, bacteria) in solution. The amount of light scattered at 90° to the incident light is directly proportional to the turbidity.

Turbidity is measured in nephelometric turbidity units (NTUs). These units of measurement are based on the amount of light scattered by particles of a polymer reference standard called

formazin. Formazin, a mixture of hydrazine sulfate and hexamethylenetetramine, produces particles which scatter light in a reproducible manner.

The Hach 4000 NTU Formazin Cartridge, when used with the Hach Digital Titrator, offers a quick, yet accurate method for the preparation of formazin standards used in turbidimeter calibration.

REQUIRED REAGENTS

Description	Unit	Cat. No.
Formazin Titration Cartridge, 4000 NTU	each	2461-01
Water, deionized	4 L	272-56

REQUIRED APPARATUS

Clippers, for opening pillows	each	968-00
Digital Titrator	each	16900-01
Filter Holder, 47 mm, magnetic base	each	13529-00
Aspirator, vacuum pump, poly	each	2131-00
Filters, membrane, 0.45 microns	100/pkg	13530-00
Flask, filtering, 1000 mL	each	546-53
Flask, volumetric, Class A, 50 mL	each	14574-41
Flask, volumetric, Class A, 100 mL	each	14574-42
Flask, volumetric, Class A, 500 mL	each	14574-49
Stopper, rubber, No. 7, one hole	6/pkg	2119-07
Tubing, rubber, 2.4 mm ID	12 ft	560-19
Tweezers, plastic	each	14282-00

OPTIONAL APPARATUS

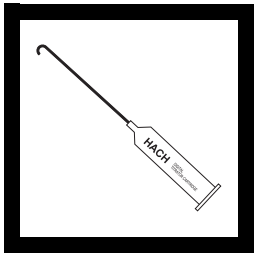
Bottle, wash poly, 500 mL	each	620-11
Clamp, 2-prong, extension, 38 mm	each	21145-00
Clamp Holder	each	326-00
Demineralizer Assembly, 473 mL	each	21846-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

VOLATILE ACIDS

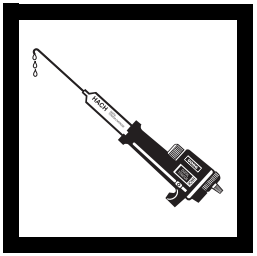
Using Sodium Hydroxide

1. Distill the sample and collect 150 mL of distillate.

Note: *Use the Volatile Acids Procedure, Sample Distillation, accompanying the General Purpose Distillation Apparatus Set or the distillation procedure described in Standard Methods for the Examination of Water and Wastewater.*



2. Attach a clean delivery tube to a 0.9274 N Sodium Hydroxide titration cartridge. Attach the cartridge to the titrator body. See *General Description, Step-by-Step,* for assembly instructions, if necessary.



3. Flush the delivery tube by turning the delivery knob to eject a few drops of titrant. Reset the counter to zero and wipe the tip.

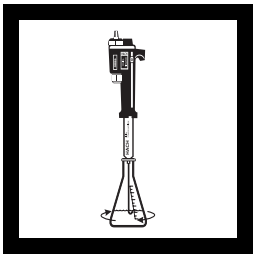
Note: *For added convenience use the TitraStir® Stir Plate. See General Description, Step 3 in Step-by-Step.*

4. Select the distillate volume corresponding to the expected volatile acids concentration as acetic acid from *Table 1*. Using a graduated cylinder, transfer the distillate volume into a clean 250-mL Erlenmeyer flask and dilute to about the 150-mL mark with deionized water.

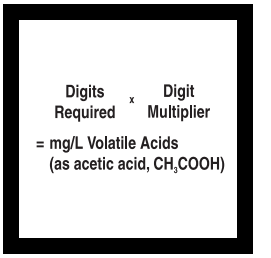
Table 1

Range (mg/L as CH_3COOH)	Volume (mL)	Titration Cartridge (N NaOH)	Catalog Number	Digit Multiplier
100-400	150	0.9274	14842-01	1
200-800	75	0.9274	14842-01	2
600-2400	25	0.9274	14842-01	6

5. Add the contents of one Phenolphthalein Indicator Powder Pillow and swirl to mix.



6. Place the delivery tube tip into the solution and swirl while titrating with sodium hydroxide until a light pink color appears. Record the number of digits required.



7. Calculate:

Digits Required x Digits Multiplier = mg/L Volatile Acids (as acetic acid, CH_3COOH)

Note: *Approximately 70% of the volatile acids in the sample will be found in the distillate. This has been accounted for in the calculation.*

Summary of Method

A sample acidified with sulfuric acid is distilled and the distillate titrated to the phenolphthalein end point with sodium hydroxide standard.

REQUIRED REAGENTS

Volatile Acids Reagent Set (about 100 tests) 24602-00
Includes: (1) 942-99, (1) 14842-01

Description	Unit	Cat. No.
Phenolphthalein Indicator Powder Pillows	100/pkg	942-99
Sodium Hydroxide Titration Cartridge, 0.9274 N	each	14842-01
Water, deionized	4 L	272-56

REQUIRED APPARATUS

Cylinder, graduated, 250 mL	each	508-46
Digital Titrator	each	16900-01
Flask, Erlenmeyer, 250 mL	each	505-46

OPTIONAL APPARATUS

Clamp, 2-prong, extension, 38 mm	each	21145-00
Clamp Holder	each	326-00
Delivery Tubes, with 180° hook	5/pkg	17205-00
Delivery Tubes, 90° with hook	5/pkg	41578-00
Distillation Heater and Support Apparatus, 115 Vac	each	22744-00
Distillation Heater and Support Apparatus, 230 Vac	each	22744-02
Distillation Apparatus Set	each	22653-00
Standard Methods for the Examination of Water and Wastewater, 19th ed.	each	22708-00
Support Ring Stand	each	563-00
TitraStir® Stir Plate, 115 Vac	each	19400-00
TitraStir® Stir Plate, 230 Vac	each	19400-10

APPENDIX A
ACCURACY CHECK AND STANDARD ADDITIONS

Most of the procedures in this manual have an accuracy check based on the standard additions method. Standard additions is a widely accepted technique for checking the validity of test results. Also known as “spiking” and “known additions,” the technique adds a small amount of the component (parameter) being measured to an analyzed sample and the analysis is repeated. The increase in the test results should equal the amount of the standard added. The results can be used to check the performance of the reagents, the apparatus, and the procedure.

First Step - The Accuracy Check

Perform the procedure and accuracy check as described in this manual. In each accuracy check the number of digits expected for each increment is given. If the actual number of digits required is within 1% of the expected number of digits, the analyst can conclude the answer for the sample is accurate and the reagents, apparatus, and method used are working properly.

Second Step - The Decision Tree

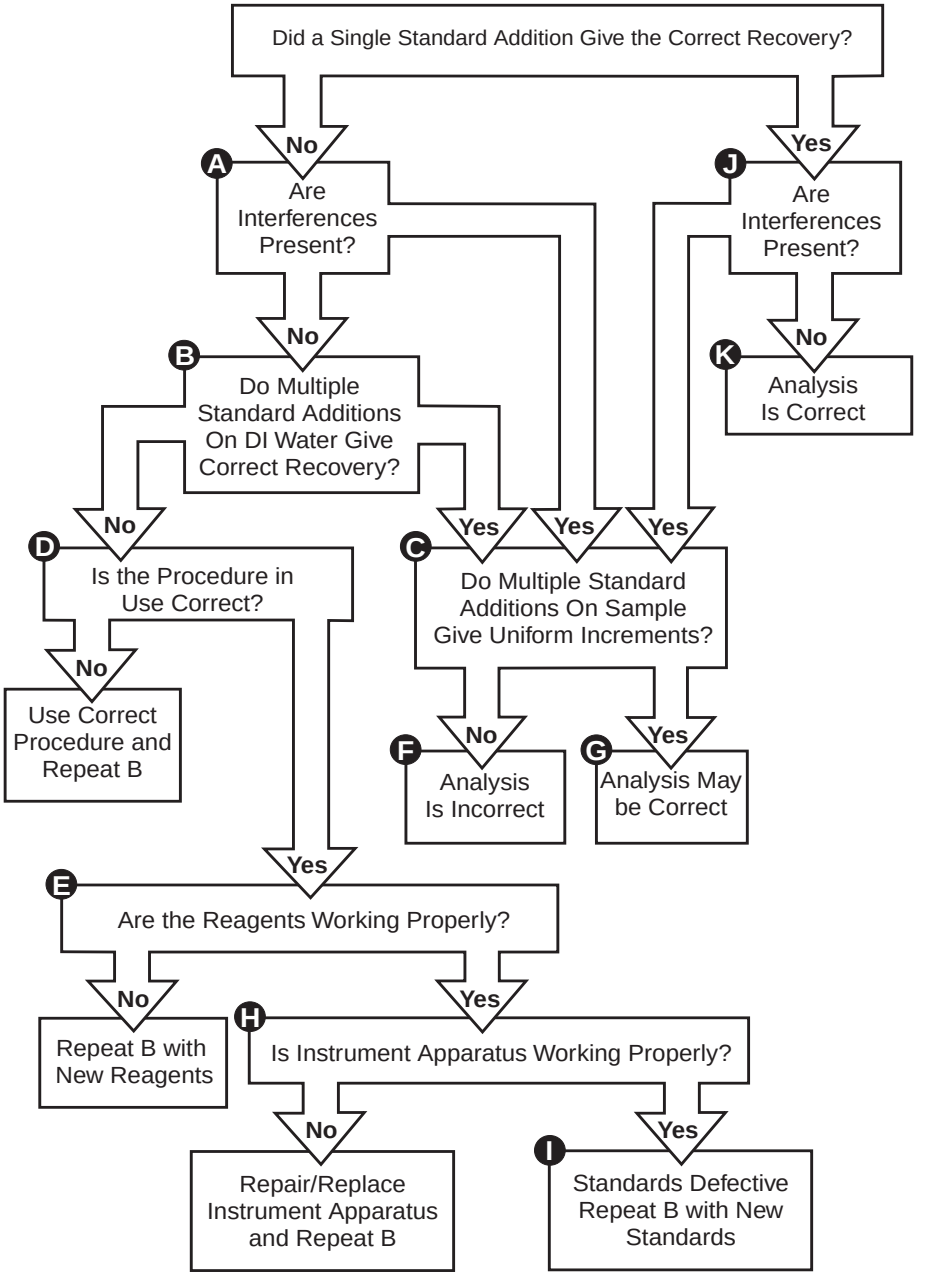
If the actual number of digits varies noticeably, then it must be concluded the problem was caused by either the reagents, the apparatus, the procedure or an interfering substance. By following a logical troubleshooting approach, the source of the problem can be systematically determined. Using the step-by-step decision tree in *Figure 1* will greatly ease identifying the problem. An explanation of each step in the decision tree follows.

Third Step - The Branches

Branch A

Suppose the first, or all three standard additions to the sample did not give the correct incremental digit increase. A possible cause for this could be the presence of interferences. Other causes could be defective reagents, an incorrect procedure, defective apparatus or a defective standard used for standard addition. If interferences are either absent or assumed to be absent, proceed to Branch B. If interferences are present, proceed to Branch C. The Chloride Procedure, Silver Nitrate Method, is used as an example throughout these steps.

Figure 1
Decision Tree



Branch B

Repeat the Accuracy Check given in the procedure substituting the same volume of deionized water for the sample. For example, using the Chloride Procedure, Silver Nitrate Method:

1. Take a 50.0-mL sample of deionized water and follow the Chloride Procedure, Silver Nitrate Method. Record the number of digits required for the titration.

2. Add 0.10 mL of Chloride Standard Solution, 12,500 mg/L, and titrate to the end point. Record the number of digits required for the titration.

3. Repeat, using two more additions of 0.1 mL of 12,500 chloride standard, titrating to the end point after each addition. Record the number of digits required.

4. Tabulate the date as shown below:

Table 1

Total Standard Added (mL)	Total Number of Digits Used	Total Standard Added (mg/L)	Total Parameter Found (mg/L)
0	_____	0	_____
0.1	_____	_____	_____
0.2	_____	_____	_____
0.3	_____	_____	_____

The **Total Standard Added (mL)** will vary depending on the procedure used.

The **Total Number of Digits Used** are the total digits recorded after each titration.

The **Total Standard Added (mg/L)** is determined for each addition by the following equation:

$$\frac{\text{Total Standard Added (mL)}}{\text{Sample Volume (mL)}} \times \text{Standard Concentration (mg/L)} = \text{Total Standard Added (mg/L)}$$

The **Total Parameter Found (mg/L)** is determined by following the calculation step of the procedure used. Use the same volume of

deionized water as used for the sample. The addition of standard will not change the digit multiplier.

Performing the above procedure, the completed table would look like this:

Table 2

Total Standard Added (mL)	Total Number of Digits Used	Total Standard Added (mg/L)	Total Parameter Found (mg/L)
0	0	0	0
0.1	25	25	25
0.2	50	50	50
0.3	75	75	75

To complete the table the following calculations were made based on the above formula:

First Addition

$$\frac{0.1}{50} \times 12{,}500 = \text{Total Standard Added (mg/L)} = 25 \text{ mg/L}$$

Second Addition

$$0.1 + 0.1 = 0.2 \text{ mL} = \text{Total Standard Added (mL)}$$

0.2 mL in the above formula gives 50 mg/L total standard added.

Third Addition

$$0.1 + 0.1 + 0.1 = 0.3 \text{ mL} = \text{Total Standard Added (mL)}$$

0.3 mL in the above formula gives 75 mg/L total standard added.

The data shown above reveals several points:

- The chemicals, apparatus, procedures and standards are in good working condition. This conclusion is made because chloride added to the deionized water sample was recovered entirely in the same uniform steps of addition.
- Because chloride added to deionized water was recovered, but was not recovered during the Accuracy Check, one may

conclude the sample contains interferences which prevent the test reagents from operating properly.

- The first analysis of the water sample gave an incorrect result.

If the above results gave the expected increments between additions, proceed to Branch C. If the results did not give the expected increments, proceed to Branch D.

Branch C

If interfering ions are present, it may be concluded the analysis is incorrect. However, with the completed accuracy check it may be possible to arrive at an approximation of the correct result. Tabulate the results as follows:

Table 3

Total Standard Added (mL)	Total Number of Digits Used	Total Standard Added (mg/L)	Total Parameter Found (mg/L)
0	_____	0	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

The **Total Standard Added (mL)** will vary depending on the procedure used.

The **Total Number of Digits Used** are the total digits recorded after each addition of standard as specified in the accuracy check.

The **Total Standard Added (mg/L)** is determined for each addition by the following equation:

$$\frac{\text{Total Standard Added (mL)}}{\text{Sample Volume (mL)}} \times \text{Standard Concentration (mg/L)} = \text{Total Standard Added (mg/L)}$$

The **Total Parameter Found (mg/L)** is determined by following the calculation step of the procedure used. Use the same volume of deionized water as used for the sample. The addition of standard will not change the digit multiplier.

If steps between each addition are roughly uniform (i.e., 25 digits or 25 mg/L difference between each addition), proceed to Branch

G. If the results are not uniform (i.e., 13, 10, and 6 mg/L), proceed to Branch F.

For example, a sample of water was analyzed for chloride with the result being 100 mg/L. The analyst, suspecting interferences, made one standard addition of 0.10 mL of 12,500-mg/L chloride standard to 50.0 mL of sample. Rather than an increase of 25 mg/L as expected, the analyst found an increase of 13 mg/L.

The analyst added a second and third addition of 0.1 mL of standard. The titrations were made and the results tabulated. The increments were 10 (123 minus 113) and 6 (129 minus 123) mg/L, respectively. The analyst proceeded to Branch F.

Table 4

Total Standard Added (mL)	Total Number of Digits Used	Total Standard Added (mg/L)	Total Parameter Found (mg/L
0	100	0	100
0.1	113	25	113
0.2	123	50	123
0.3	129	75	129

To complete the table the following calculations were made based on the above formula:

First Addition

$$\frac{0.1}{50} \times 12{,}500 = \text{Total Standard Added (mg/L)} = 25 \text{ mg/L}$$

Second Addition

$$0.1 + 0.1 = 0.2 \text{ mL} = \text{Total Standard Added (mL)}$$

0.2 mL in the above formula gives 50 mg/L Total Standard Added.

Third Addition

$$0.1 + 0.1 + 0.1 = 0.3 \text{ mL} = \text{Total Standard Added (mL)}$$

0.3 mL in the above formula gives 75 mg/L Total Standard Added.

Branch D

Carefully check the instructions or directions for use of the procedure, making sure the proper techniques, reagents, titrant, sample volume, and digit multiplier were used. Verify there is no air or liquid, other than the titrant being used, in the delivery tube by ejecting several drops of solution. If the procedure in use is found to be in error, repeat Branch B using the correct procedure. If the procedure is found to be correct, proceed to Branch E.

Branch E

Check the performance of the reagents. This may be done easily by using a known standard solution to run the test or by obtaining a new fresh lot of the reagent. A list of known standard solutions is given in *Table 1* on page *20*. If it is determined reagents are defective, repeat Branch B with new reagents. If the reagents are proven in good condition, proceed with Branch H.

Branch F

Examples of non-uniform increments between standard additions on a sample are shown below in *Table 5*, *Table 6* and *Figure 2* on page 180. These plots illustrate the effect of interferences upon the standard addition and upon substances in the sample. The plots were made by graphing the Total Standard Added (mg/L) on the X axis and the Total Parameter Found (mg/L) on the Y axis as shown in *Figure 2* on page 180.

Table 5 Plot A

Total Standard Added (mL)	Total Number of Digits Used	Total Standard Added (mg/L)	Total Parameter Found (mg/L)
0	100	0	100
0.1	113	25	113
0.2	123	50	123
0.3	129	75	129

Table 6 Plot B

Total Standard Added (mL)	Total Number of Digits Used	Total Standard Added (mg/L)	Total Parameter Found (mg/L)
0	0	0	0
0.1	25	25	0
0.2	50	50	25
0.3	75	75	50

Both of these plots show that the four data points do not lie on a straight line. Plot A illustrates an interference which becomes progressively worse as the concentration of the standard increases. This type of interference is not common and may be caused by an error or malfunction of the procedure, reagents or apparatus. Perform Branch B to ensure that the supposed interference is present.

Plot B illustrates a common chemical interference which becomes less or even zero as the concentration of the standard increases. The plot shows the first standard addition was consumed by the interference and the remaining additions gave the correct increase of 25 mg/L for each additional 0.1 mL of standard added. The apparent interference in Plot B could be the result of an error made in the standard addition, and the analysis should be repeated with a fresh portion of sample.

The two examples illustrate chemical interferences which most certainly mean the result of the first analysis of the water sample was incorrect. When this type of interference is encountered, review the Interference section for the procedure for corrective steps. If this fails, the analyst should attempt to analyze the sample with an alternate method which, if possible, uses a different type of chemistry.

Branch G

Examples of uniform increments between standard additions on a sample are shown below in *Table 7* and *Table 8* on page 179. These plots illustrate the effect of interferences upon the standard addition and upon substances in the sample. The plots were made by graphing the Total Standard Added (mg/L) on the X axis and the Total Parameter Found (mg/L) on the Y axis as shown in *Figure 2* on page 180.

Table 7 Plot C

Total Standard Added (mL)	Total Number of Digits Used	Total Standard Added (mg/L)	Total Parameter Found (mg/L)
0	50	0	50
0.1	63	25	63
0.2	75	50	75
0.3	88	75	88

Plot C illustrates a common interference with a uniform effect upon the standard and the substances in the sample. The four data points form a straight line, but the titration increments between the additions is not correct. The straight line between the additions may be extrapolated back through the horizontal axis. The point of intersect of the line with the horizontal axis gives a more accurate estimate of the concentration of the substance in question for the sample. In the example, the first analysis of the sample gave 50 mg/L. The result located graphically (100 mg/L) using the accuracy check should be much closer to the correct result. Other interference effects may also be present, or interferences may not be consistent in all samples. Use of another method not subject to the interference, or elimination of the interference is preferred over extrapolation or use of the percent recovery calculation.

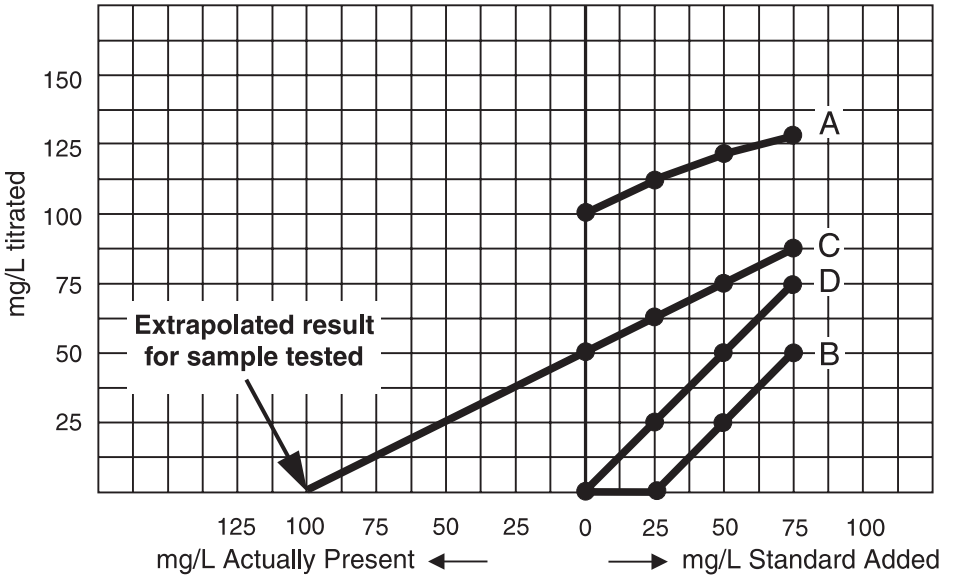
Apparent interferences also may be caused by errors in the method, a defect in the apparatus or standards. Before assuming the interference is chemical in nature, perform Branch B.

Table 8 Plot D

Total Standard Added (mL)	Total Number of Digits Used	Total Standard Added (mg/L)	Total Parameter Found (mg/L)
0	0	0	0
0.1	25	25	25
0.2	50	50	50
0.3	75	75	75

Plot D illustrates correct results but may hide a problem for the analyst. The increments found are uniform and the recovery of the standard was complete. The result of the first analysis was 0 mg/L and the plot graphs back through 0 mg/L. If interfering species are present, the interference may be sufficient to change the sample result but not sufficient to prevent the analyst from finding uniform increments and complete recovery of the additions. This would be an uncommon situation and results are probably correct unless unusual interferences are possible. Refer to the Interferences section in the specific procedure.

Figure 2
Multiple Standard Additions Graph



Branch H

Check operation of the apparatus used in the performance of the test. Verify the correct volumes of sample and standard were used. Check glassware used in the procedure, making sure that it is scrupulously cleaned. Dirty pipets and graduated cylinders are a source of contamination and will not deliver the correct volume. If a defect is found in the apparatus, repeat Branch B after repair or replacement of apparatus. If the apparatus is found to be in good working order, proceed with Branch I.

Branch I

After demonstrating that the procedure, reagents, and apparatus are correct and operating properly, the only possible cause for standard additions not functioning properly in deionized water is the standard used in performing the standard additions. Prepare or obtain a new set of standards and repeat Branch B.

Branch J
If the standard addition gave the correct result, the analyst must then determine if interfering substances are present. If interfering substances are not present, the result of the analysis prior to the standard addition is correct. If interfering substances are present, proceed to Branch C.

One of the greatest aids to the analyst is knowledge of the water sample's composition. An analyst need not know the exact composition of each sample but should be aware of potential interferences in the method of analysis to be used. When performing a particular method, the analyst should know if those interferences are present or not in order to have confidence in the accuracy of the results. Once the interferences are known, the Interference section of each procedure describes how to correct for many common interferences.

If the correct result is obtained with one standard addition when no interfering species are present, the chance of an error in sample results is very small. Possible sources of error not revealed include: sample quality, sample quantity (unless the sample and standard volume used is equal), and inconsistent end point determinations.

Call Hach Technical and Customer Support (1-800-227-4224) for additional help.

GENERAL INFORMATION

At Hach Company, customer service is an important part of every product we make.

With that in mind, we have compiled the following information for your convenience.

REPLACEMENT PARTS AND ACCESSORIES

REPLACEMENT PARTS FOR MODEL 16900 DIGITAL TITRATOR

Description	Unit	Cat. No.
Digital Titrator including delivery tubes, manual, and case	each	16900-01
Delivery Tubes, 180° hook	5/pkg	17205-00
Case	each	46602-00
Digital Titrator Manual	each	16900-08

HOW TO ORDER

By Telephone:

6:30 a.m. to 5:00 p.m. MST
Monday through Friday
(800) 227-HACH
(800-227-4224)
By FAX: (970) 669-2932

By Mail:

Hach Company
P.O. Box 389
Loveland, CO 80539-0389
U.S.A.

Ordering information by E-mail: orders@hach.com

Information Required

- Hach account number (if available)
- Your name and phone number
- Purchase order number
- Brief description or model number
- Billing address
- Shipping address
- Catalog number
- Quantity

Technical and Customer Service (U.S.A. only)

Hach Technical and Customer Service Department personnel are eager to answer questions about our products and their use. Specialists in analytical methods, they are happy to put their talents to work for you.
Call **1-800-227-4224** or E-mail **techhelp@hach.com**.

International Customers

Hach maintains a worldwide network of dealers and distributors. To locate the representative nearest you, send E-mail to **intl@hach. com** or contact:

In Canada, Latin America, Africa, Asia, Pacific Rim:
Telephone: (970) 669-3050; FAX: (970) 669-2932

In Europe, the Middle East, or Mediterranean Africa:
HACH Company, c/o
Dr. Bruno Lange GmbH
Willstätterstr. 11
D-40549 Düsseldorf
Germany
Telephone: +49/[0]211.52.88.0
Fax: +49/[0]211.52.88.231

REPAIR SERVICE

Authorization must be obtained from Hach Company before sending any items for repair. Please contact the HACH Service Center serving your location.

In the United States:
Hach Company
100 Dayton Avenue
Ames, Iowa 50010
(800) 227-4224 (U.S.A. only)
Telephone: (515) 232-2533
FAX: (515) 232-1276

In Canada:
Hach Sales & Service Canada Ltd.
1313 Border Street, Unit 34
Winnipeg, Manitoba
R3H 0X4
(800) 665-7635 (Canada only)
Telephone: (204) 632-5598
FAX: (204) 694-5134
E-mail: canada@hach.com

In Latin America, the Caribbean, the Far East, the Indian Subcontinent, Africa, Europe, or the Middle East:
Hach Company World Headquarters
P.O. Box 389
Loveland, Colorado, 80539-0389
U.S.A.
Telephone: (970) 669-3050
FAX: (970) 669-2932
E-mail: intl@hach.com

WARRANTY

Hach warrants most products against defective materials or workmanship for at least one year from the date of shipment; longer warranties may apply to some items.

HACH WARRANTS TO THE ORIGINAL BUYER THAT HACH PRODUCTS WILL CONFORM TO ANY EXPRESS WRITTEN WARRANTY GIVEN BY HACH TO THE BUYER. EXCEPT AS EXPRESSLY SET FORTH IN THE PRECEDING SENTENCE, HACH MAKES NO WARRANTY OF ANY KIND WHATSOEVER WITH RESPECT TO ANY PRODUCTS. HACH EXPRESSLY DISCLAIMS ANY WARRANTIES IMPLIED BY LAW, INCLUDING BUT NOT BINDING TO ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE.

LIMITATION OF REMEDIES: Hach shall, at its option, replace or repair nonconforming products or refund all amounts paid by the buyer. **THIS IS THE EXCLUSIVE REMEDY FOR ANY BREACH OF WARRANTY.**

LIMITATION OF DAMAGES: IN NO EVENT SHALL HACH BE LIABLE FOR ANY INCIDENTAL OR CONSEQUENTIAL DAMAGES OF ANY KIND FOR BREACH OF ANY WARRANTY, NEGLIGENCE, ON THE BASIS OF STRICT LIABILITY, OR OTHERWISE.

This warranty applies only to Hach products purchased and delivered in the United States.

Catalog descriptions, pictures and specification, although accurate to the best of our knowledge, are not a guarantee or warranty.

For a complete description of Hach Company's warranty policy, request a copy of our Terms and Conditions of Sale for U.S. Sales from our Customer Service Department.

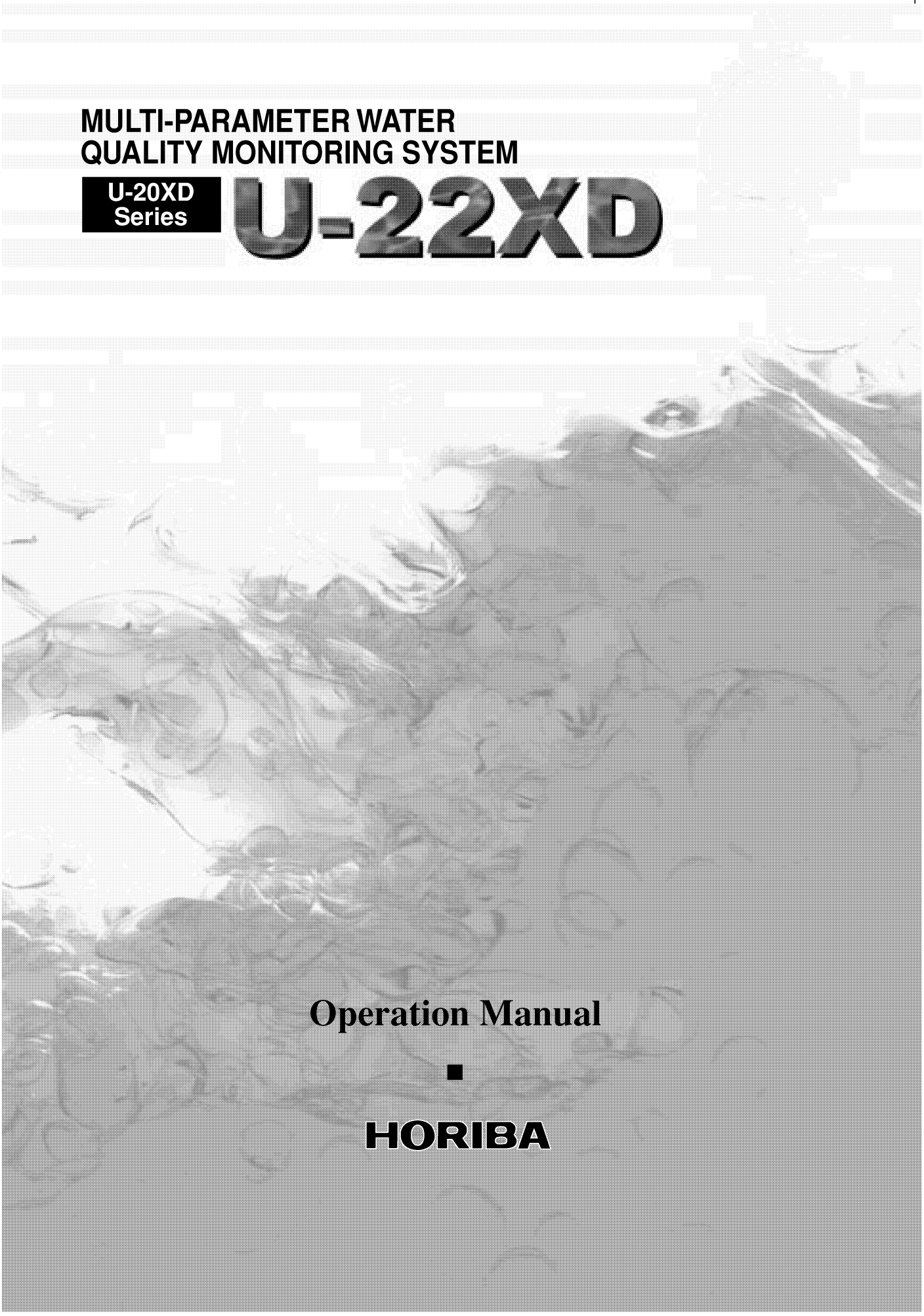
HACH COMPANY
WORLD HEADQUARTERS
P.O. Box 389
Loveland, Colorado 80539-0389
Telephone: (970) 669-3050
FAX: (970) 669-2932

FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. - **Call toll-free 800-227-4224**
Outside the U.S.A. - **Contact the HACH office or distributor serving you.**
On the Worldwide Web - **www.hach.com**; E-mail - **techhelp@hach.com**

MULTI-PARAMETER WATER QUALITY MONITORING SYSTEM

U-20XD Series

U-22XD



Operation Manual

■

HORIBA

HORIBA's Warranty and Responsibility

Your U-20XD series multi-parameter water quality monitoring system is covered by HORIBA's warranty for a period of one (1) year, under normal use. Although unlikely, if any trouble attributable to HORIBA should occur during this period, necessary exchange or repairs shall be conducted by HORIBA, free of charge.

The warranty does not cover the following:

- Any trouble or damage attributable to actions or conditions specifically mentioned in the operation manuals to be avoided
- Any trouble or damage attributable to use of the multi-parameter water quality monitoring system in ways or for purposes other than those described in the operation manuals
- If any repairs renovations, disassembly, etc. are performed on this multi-parameter water quality monitoring system by any party other than HORIBA or a party authorized by HORIBA
- Any alteration to the external appearance of this multi-parameter water quality monitoring system attributable to scratches, dirt, etc. occurring through normal use
- Wear and tear to parts, the exchange of accessories, or the use of any parts not specified by HORIBA

INSTALLATION ENVIRONMENT

This product is designed for the following environment:

→ Installation Categories II

→ Pollution degree 2

LIMITATION OF LIABILITY FOR DAMAGES

HORIBA will not accept responsibility for damage or malfunction that may occur as a result of operation or situation not recommended in this manual. HORIBA shall not be liable for Customer's incidental, consequential or special damages, or for lost profits or business interruption losses, in connection with the operation of the Manufactured Parts, CPU hardware, disk drives or Software.

CE MARKING

CE

U-20XD Series conforms with the following directive(s) and standard(s):

Directives:

the EMC Directive 89/336/EEC, in accordance with Article 10(1) of the Directive

the Low Voltage Directive 73/23/EEC

Standards:

[the EMC Directive] EN61326:1997+A1:1998
(EMISSION : Class B, IMMUNITY Category : General)

[the Low Voltage Directive] EN61010-1:1993 +A2:1995

FCC 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 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.

Unauthorized reprinting or copying of this operation manual

No unauthorized reprinting or copying of all or part of this operation manual is allowed. The utmost care has been used in the preparation of this operation manual. If, however, you have any questions or notice any errors, please contact the HORIBA customer service printed on the back cover of this operation manual.

Preface

Thank you very much for purchasing HORIBA's "MULTI-PARAMETER WATER QUALITY MONITORING SYSTEM" U-20XD Series.

Compact and one-hand-held, our multi-parameter water quality monitoring system makes measurements about a large number of items simultaneously.

The instrument uses a large-sized LCD display and has a variety of functions through easy operation, being useful for use at sites where measurements are to be made.

The water-proof construction of the instrument is compliant with IP-67 of IEC 529, "Water-proof test on electrical and mechanical equipment and tools and protection grade against entry of solids." Please use the instrument by following the information in this Operation Manual to maintain the water-proof construction of the instrument.

IP-67 standards

- Keeping dust and grit out of the instrument
- Up to 5 °C difference between water and an instrument employed and no entry of water into. the inside of the instrument at a depth of 1 m for 30 minutes

This Operation Manual contains information on the basic way of handling the instrument, notes, etc. for the user. Be sure to read through the Operation Manual before use.

Symbols employed

The symbols employed herein have the following meanings:

⚠WARNING : Improper use can result in serious injury or even death.

⚠CAUTION : The improper use of the instrument may cause the following dangers:

- Danger of injury
- Danger of damage to the instrument, its peripherals, and data

: Description of what should never be done, or what is prohibited.

: Description of what should be done, or what should be followed.

Important : Explanation necessary for the proper operation of the instrument

Note : Explanation that is useful and necessary for handling the instrument

☞ : Refer to the item shown.

Symbols employed in screen description

The symbols and used in screen description have the following meanings:

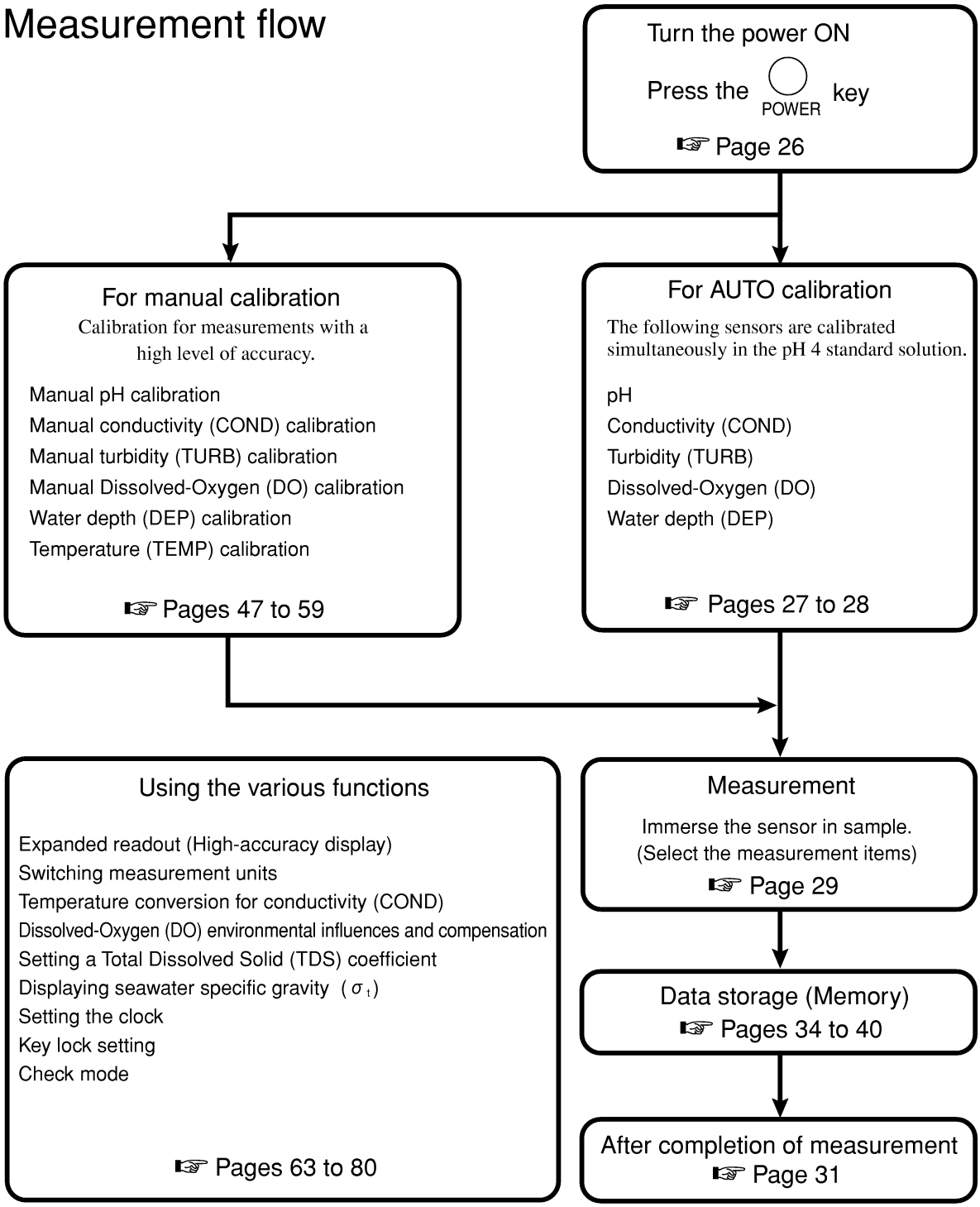
: The letters and numbers in this symbol are blinking on the screen.

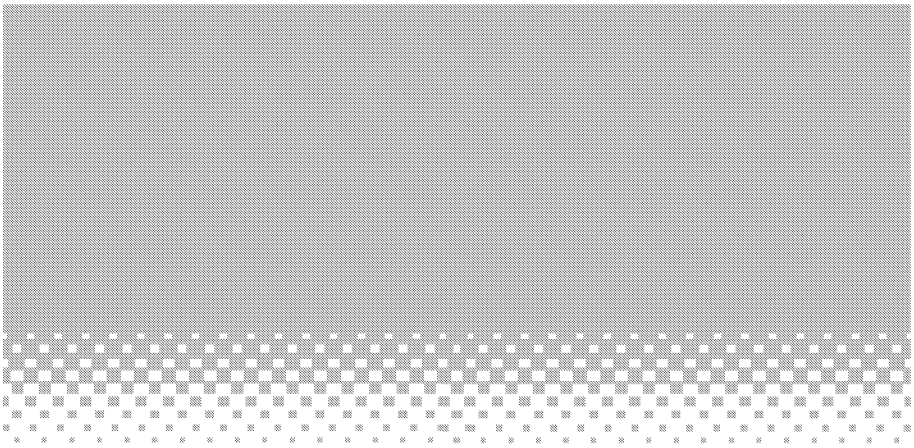
: The letters and numbers in this symbol are lighting up on the screen.

◍ CONTENTS ◍

◍ CONTENTS ◍

Measurement flow



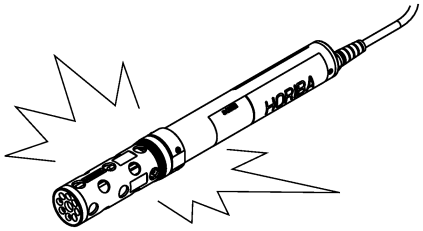


1. Introduction

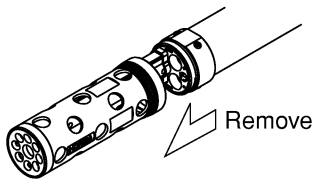
1.1 Notes on handling the instrument

Handling of sensor probe

Do not give a shock to the sensor probe.
The sensor will be damaged.

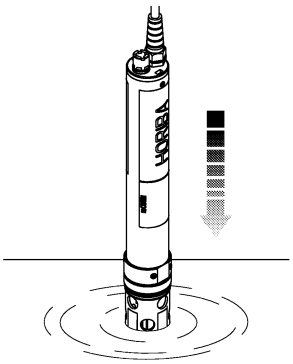


Do not remove the protection cover from the sensor probe to use.
Damage may occur to the sensor.



Slowly lower the sensor probe into the sample.

Dropping it from a height of 1m or more may cause damage to the sensor.
Do not immerse the sensor probe at the depth of exceeding 100 m.
The device can resist the hydraulic pressure at the dept up to 100 m.



- The protection cover may rust due to the environment in which it is used. The damage caused by this usage shall not be warranted by the manufacturer. Solve it with parts which users need to replace periodically.

⚠WARNING

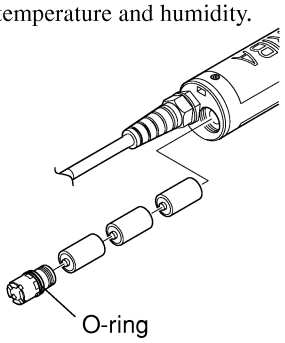
- Fix the sensor probe to the cable or the reel to use.
- In place with a large distance to the water level or with a rapid water flow, fix the sensor probe hook to a point except your body before use for safety purposes.
  Be careful not to let go off the sensor probe by mistake. Otherwise, the sensor probe together with the instrument will fall into the water or a sharp shock will occur to yourself while you are holding the instrument.

Replacing batteries and sensor of the sensor probe

Do not replace the sensor probe batteries and sensor in the atmosphere of high temperature and humidity.

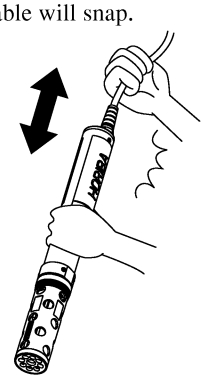
Put connector plugs into the sensor probe connectors with sensors off.

The sensor probe's battery cover is kept waterproof by the use of an O-ring. After checking that there are no foreign bodies adhering to the O-ring, apply silicon grease (included) to the face of the O-ring and close. Be sure to close it all the way to the indicated level. Do not close with the O-ring twisted or warped.

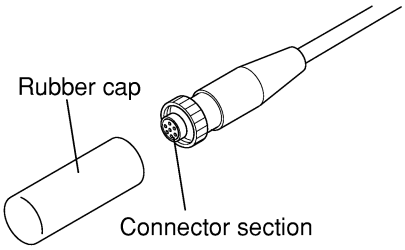


Handling of cable

Do not store the cable with its connector being greatly tensed or bent.
Do not submit the connector to strong shocks or the cable will snap.

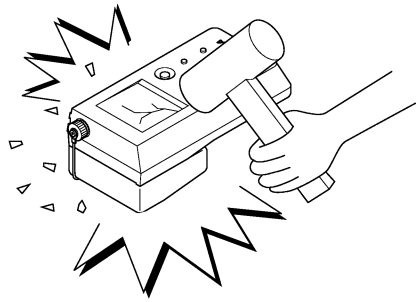


If sample waterdrops remain onto the connector section, metal part of the connector is likely to rust. When storing, wipe the area around the connector well and cover it with the rubber cap.

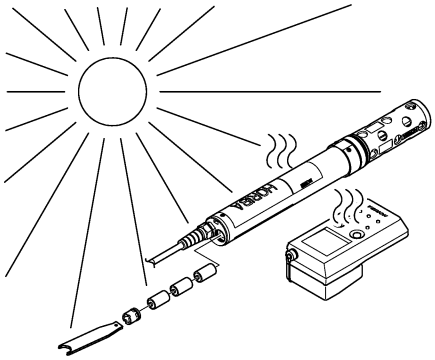


Handling of the instrument

Do not give a shock to or drop the sensor or instrument. The sensor or instrument will be damaged.

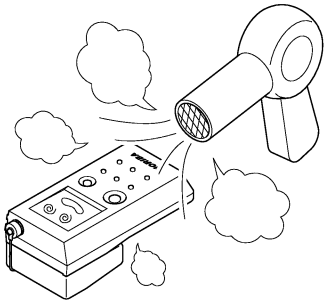


The display part includes LCD. Do not expose the instrument to ultraviolet rays for a long time. Otherwise, the LCD may deteriorate.

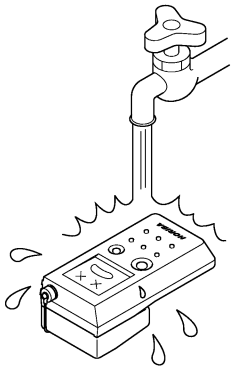


The instrument will be water-proof in construction (IP-67) when the sensor connector is connected to the instrument. However, if the instrument has been dropped into water or become wet, use a soft cloth to dry up the instrument.

Do not use a hair dryer to dry up the instrument.

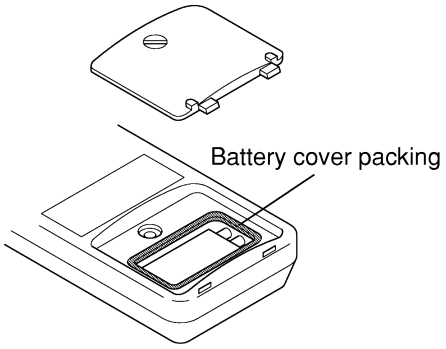


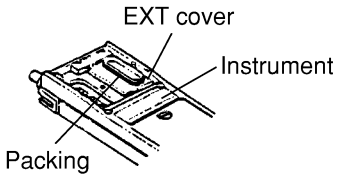
Do not wash directly the instrument using tap water from the faucet.



Note on replacing battery of the instrument and the section to which the EXT unit is attached

Waterproof function of the main unit is maintained by the packing of battery cover and EXT unit cover. Foreign matter on the packing can cause water to enter the instrument. Check for foreign matter on the packing before closing the battery cover and the EXT cover.
If the packing is twisted, do not close the battery cover and the EXT cover.





For a long use

We recommend that the packing be replaced once a year.
For battery cover packing replacement, contact your sales agent.

Note on place for use

- Avoid continuous measurement in water containing alcohol, organic solvent, strong acid, strong alkali or neutral detergent; otherwise the sensor surface will deteriorate.
- Do not use the instrument in the atmosphere with ambient temperatures below 0 °C (incl.) or above 55 °C (incl.).
- Avoid using the instrument in the condition exposed to strong vibrations or corrosive gases.
- Do not use the instrument near a source of strong electromagnetic field such as high-voltage cables and motors.

Batteries

The improper use of batteries may cause leaks and explosion.
Observe the followings:

- Set the batteries in place properly while paying attention to the plus (+) and minus (-) poles.
- Do not use both an old and new batteries or batteries of different types.
- Batteries for use in the instrument are not of the rechargeable type.
- Remove the batteries when not in use for a long time.
  In case of leaks, wipe off the solution in the battery case thoroughly and place new batteries in position.

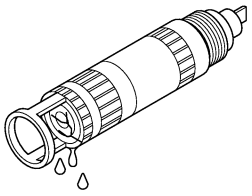
Handling the DO sensor

- In case of breakage of DO sensor diaphragm, replace DO sensor or replace just the diaphragm by using diaphragm replacement unit, without directly touching the internal solution.
- When removing the DO sensor from the sensor probe, make sure to install the short socket (included).

- Do not give a shock to the DO sensor. The sensor will be damaged.

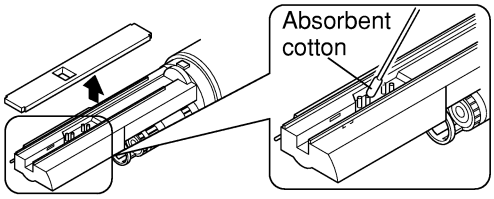
⚠CAUTION

- The DO sensor holds a strong-alkaline solution. Protect the eye and skin from the solution. If there is any solution in the eye or on the skin, immediately use sufficient water to wash off the solution. Consult a doctor as required.



Handling the COND/TURB unit

When cleaning the COND/TURB unit, use an absorbent cotton to avoid damage to the TURB cell.

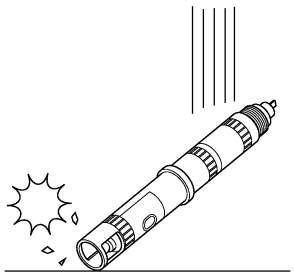


Handling the pH/ORP sensor

The pH/ORP sensor has a glass electrode at the end. Handle the sensor carefully to avoid a break in the glass electrode.

⚠CAUTION

- Be careful not to break the glass on the top of the sensor. Otherwise you may get hurt with a piece of glass.



Disposal

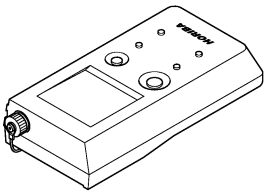
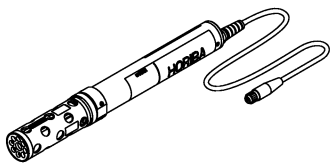
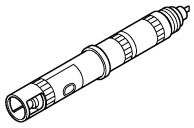
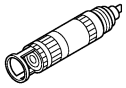
Dispose of this product as special waste, otherwise this may affect the environment.

Handling in transportation

- When transporting this product as freight, use the carrying case to prevent damage.
- Remove the flow cell from the sensor probe in transportation.

1.2 Packing list

The U-20XD series is comprised of the following items.

Model	U-22XD
Meter (U-2000)	
Sensor probe	
Sensor	pH/ORP sensor 
	DO (Dissolved-Oxygen) Sensor 
Accessories	pH 4 standard solution (500 mL)
	pH internal solution (250 mL)
	pH syringe with needle
	Sensor spanner
	Calibration beaker
	Probe cap
	Grip holder
	Carrying case
	Manganese battery 6F22 (006P) (1 piece)
	Alkaline batteries LR03 (AAA) (3 pieces)
	Silicon grease
	Sensor O-ring (S8) (10 pieces)
	Sponge for probe cap (5pieces)
	Operation manual

- The included battery is for the monitor. Its life is not guaranteed.

2. Before Use

2.1 Introduction to the instrument

2.1.1 Measurement items

Components that can be measured with the U-20XD series models are as follows:

Model	U-22XD
Measurement items	
pH	○
Dissolved Oxygen (DO)	○
Conductivity (COND)	○
Salinity (SAL) [Conductivity conversion]	○
Total dissolved solids (TDS) [Conductivity conversion]	○
Specific gravity of seawater [Conductivity conversion]	○
Temperature (TEMP)	○
Turbidity (TURB)	○
Water depth (DEP)	○
Oxidation-Reduction Potential (ORP)	○

○ ..... Measurable

2.1.2 Introduction to functions of the instrument

Outline of the functions of the instrument is described below.

Feature	Function name	Page
Data obtained during measurement can be saved in the memory.	Manual data storage	Page 34
Data can be automatically saved in the memory at constant time intervals.	Auto data storage	Page 36
Saved data can be called.	DATA OUT	Page 41
The latest date of calibration and its details can be called.	Calibration history	Page 43
Enlarged display is available.	Expand readout	Page 63
Measurement units can be switched.	Switching measurement unit	Page 64

* Other functions possible in the check mode are available. (☞ Page 73)

2.1.3 Functions of expansion units

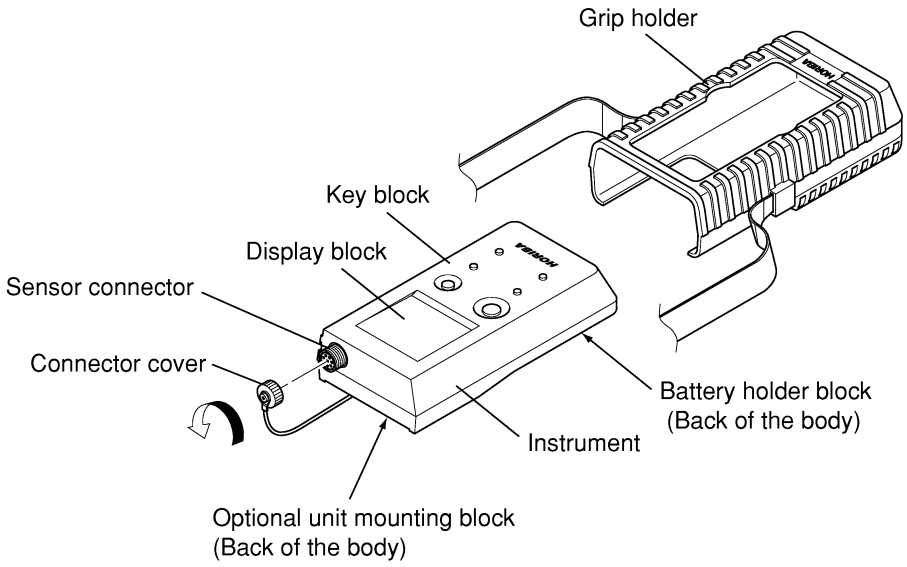
For the U-20XD series, use of expansion units allows communications with personal computers through RS-232C, the storage of G.P.S. data in the memory, and printer output, and commercial power supply.
Expansion units are available in the following two types:

Unit/name	Contents	Functions
U-2001	• Expansion adaptor	<RS-232C communications, G.P.S connection, and printer output>
Expansion adaptor	• Software for PC	The above functions cannot be used at the same time. One of the connectors for these three functions needs to be used.
U-2002	• System unit contain case	<RS-232C communications, G.P.S connection, printer output, battery power supply*>
System unit	• Software for PC	The above functions can be used at the same time.
	• G.P.S. unit	* A battery power supply can be used for measurements outdoors for 30 consecutive days.
	• Printer set	

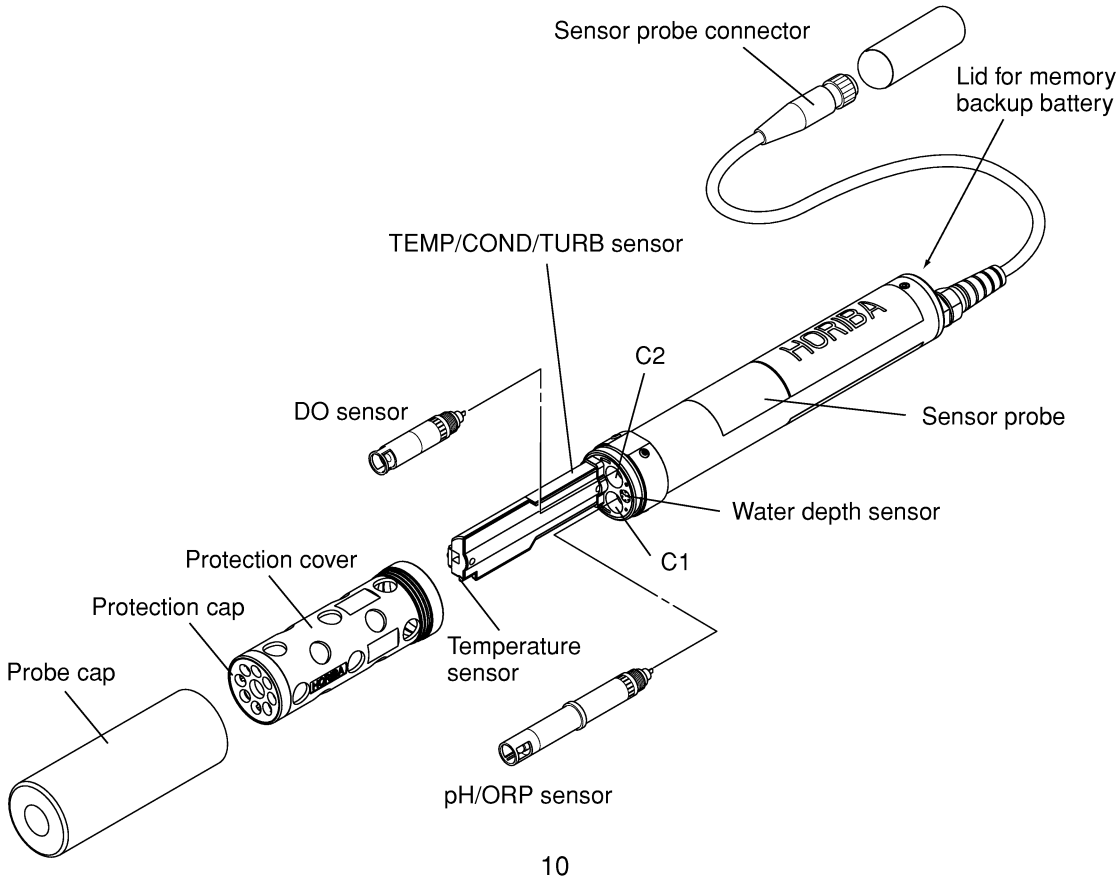
* U-2001 and U-2002 can operate on a commercial power supply through the use of an AC adapter (optional). However, the AC adapter cannot be used for the G.P.S. unit or printer set.

2.2 Names of the parts

2.2.1 Instrument name

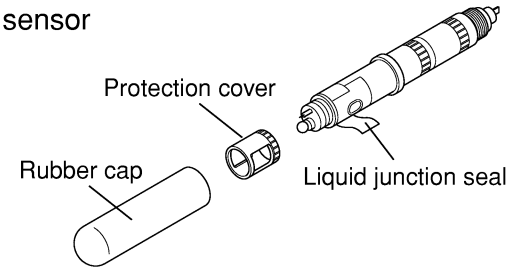


2.2.2 Sensor probe names

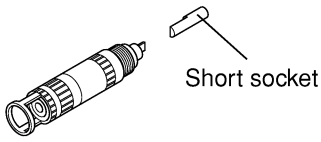


2.2.3 Sensor names

pH/ORP sensor



DO sensor



2.2.4 Use of carrying case

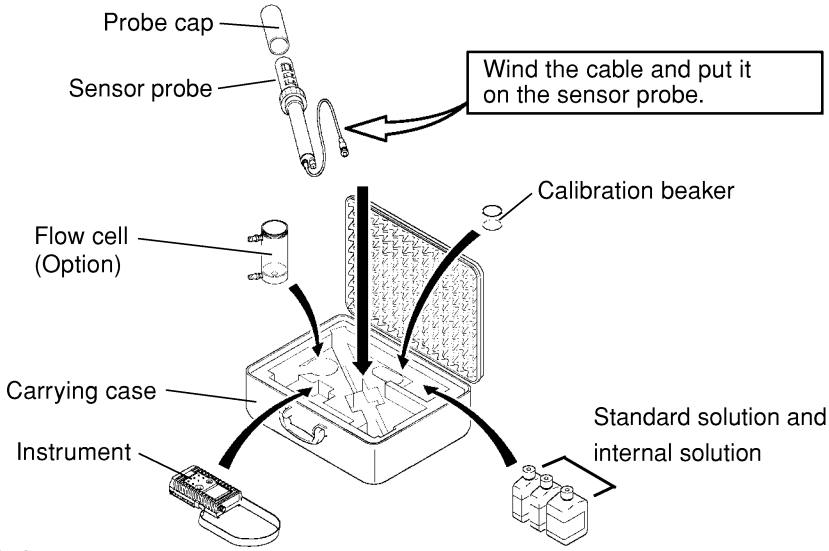
The carring case models W-2010 and W-2030 are applicable to store or transport U-22XD series.

Model	Applied to	Storage temperature	Material
W-2010	Cable length 10 m or less	-5 to 60℃	PP, ABS
W-2030	Cable length 30 m or more		

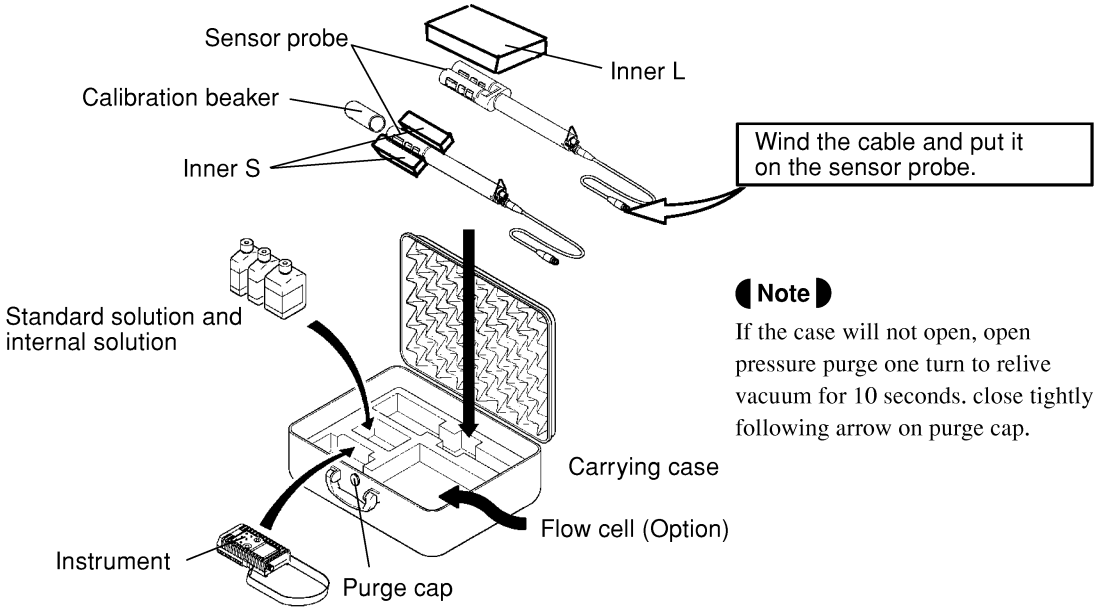
⚠CAUTION

- Do not drop or hit the carring case to protect the units against damage.
- When using the sensor probe with flow cell, separate them for strage.
- Be careful not to catch your finger, when fastening or releasing the laches.

• W-2010



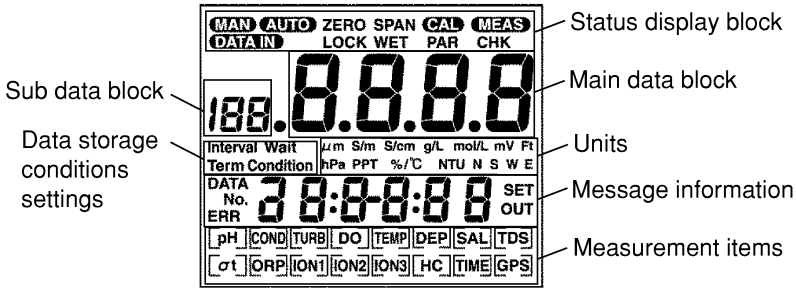
• W-2030



Note

If the case will not open, open pressure purge one turn to relive vacuum for 10 seconds. close tightly following arrow on purge cap.

2.2.5 Display



Status display block

MAN ..... On when the data memory and calibration settings are set to manual.
AUTO ..... On when the data memory and calibration settings are set to automatic.
DATA IN ..... On when the data memory operation and the data memorys operation settings are being performed. Blinking during calibration.
ZERO ..... On in the Zero calibration mode.
SPAN ..... On in the Span calibration mode.
CAL ..... On in the Calibration mode.
MEAS ..... On in the Measurement mode. (Measurements are being made when light up.)
LOCK ..... On when the keys are locked.
CHK ..... On when the instrument is in the check mode.

Sub data block

Display of the pH, Latitude (degree), Longitude (degree), Year and Check No.

Main data block

Display of Measurement data, Latitude (minute, 〔second〕), Longitude (minute, 〔second〕), and month and day.

Data storage conditions setting

Interval ..... On when a time interval is set for storage of data.
Wait ..... On when a time is set for waiting from the automatic data storage instruction until the start and during data processing through individual operations.
Term ..... On when a period is set for automatic data storage.

Units

Displays the units for measurement items.

Message information

Displays the stored data (data mode) and the data No. when the data is stored.
SET ..... Indicates that the instrument is in Set mode.

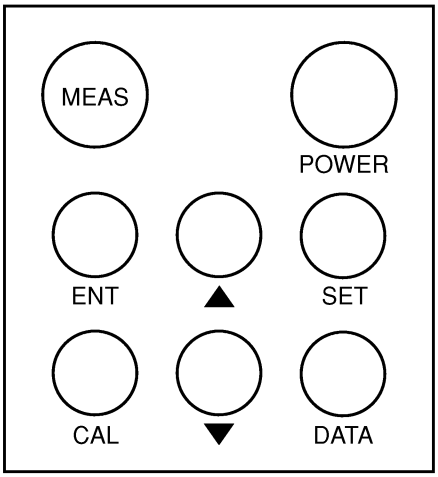
Measurement items

Displays the measurement items for the data in the main data block display. The display is read as follows.
Items without brackets ([ ]) ..... Items with the highlighted text will be stored in the data memory.
(☞ *Measurement item setting*, page 76)
Items with brackets ([ ]) ..... Displays the measurement items with data display.
(☞ *Measurement item setting*, page 76)

Note

- Because of the instrument's automatic power off function, the indication will disappear if the unit is not used for about 30 minutes. For operation of the unit and display of the indication, turn ON the instrument again.

2.2.6 Key names



POWER: Power key

Turns the instrument On and Off. Immediately after the power is switched on, the initial screen is displayed to indicate the status of the instrument.

MEAS: Measurement key

In the Measurement mode (MEAS is on), this key switches the measurement item. In addition, pressing the MEAS key returns you from the Setting, Calibration and Memory Call Up modes to the Measurement mode.

Note

- Regardless of which mode the instrument is in, it is always possible to return to the Measurement mode by pressing the MEAS key.

ENT: Enter key

In the Measurement mode (MEAS is on), pressing the ENT key stores the data in memory.
In the Calibration mode (CAL is on), pressing the ENT key performs calibration.
In the Setting mode, pressing the ENT key switches the setting and registers entered setting values.

CAL: Calibration key

Pressing the CAL key switches the instrument to the Calibration mode. If automatic data storage is in progress, it is aborted.

SET: Set key

Pressing the SET key switches the instrument from the Measurement mode to the Set mode. If the SET key is pressed on the “year, month, day, time” display screen, it switches the instrument to the Check mode.

DATA: DATA key

Pressing the DATA key switches the instrument to the Data mode.

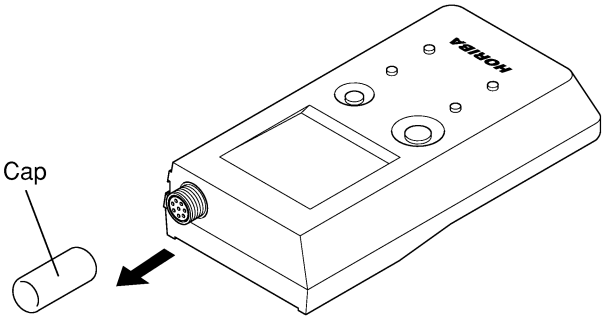
▲▼ : UP/DOWN keys

Use the UP/DOWN (▲▼) keys to set the calibration value in the Manual mode.

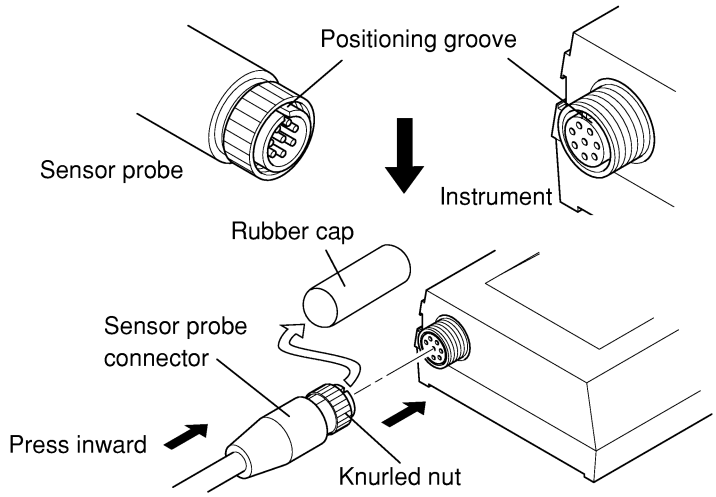
2.3 Setting up the U-20XD series models

2.3.1 Instrument and sensor probe connection

1. Remove the cap from the instrument's connector.



2. Align the positioning grooves of the instrument's connector and sensor probe connectors, and fit the connector of the sensor probe into the this other.



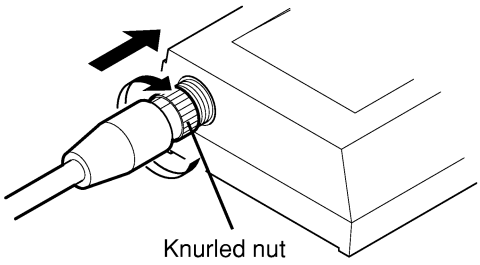
3. Press the sensor probe connector inward and turn. Tighten the connector until it will not turn any more.

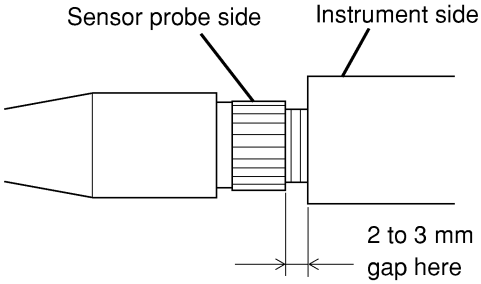
⚠CAUTION

- Turn the knurled nut with holding the knurled part. Otherwise, it will cause breaking of wire.

Important

- The connector cover or sensor probe connector should be connected to the instrument. Otherwise, the instrument will not be waterproof.
- Unless snugly attached, the instrument is not fully waterproof. When the sensor probe connector is tightened as far as it can go, a 2 to 3 mm gap is left between the instrument's connector and sensor probe connector.





Note

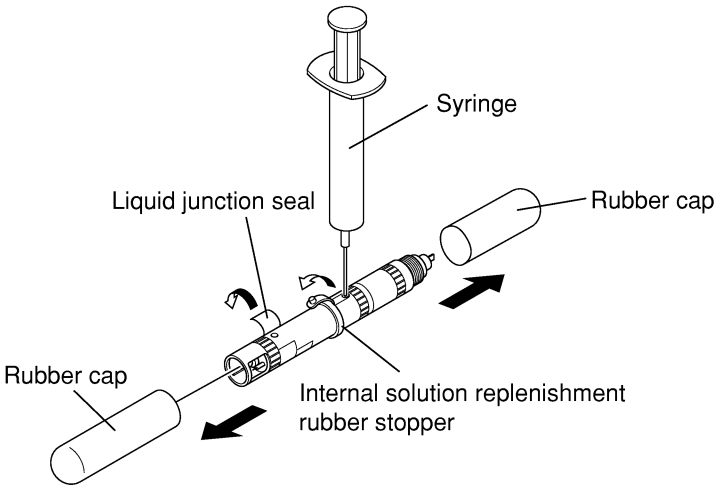
- Tighten the sensor probe connector until it will not turn any more.

2.3.2 Sensor installation

Connect the Dissolved Oxygen (DO) and pH/ORP sensors to the sensor probe.

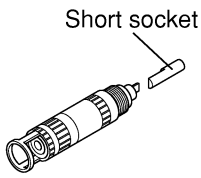
Preparing pH/ORP sensor

1. Remove the liquid junction seal and rubber caps.
2. Open the internal solution replenishment rubber stopper. Then use a syringe to take internal solution (#330).
   Air bubbles in the internal solution may impair the pressure compensation of the sensor. Allow as few air bubbles as possible to enter the inside solution.



Preparing DO sensor

1. Remove the short socket.



Important

- Provide the DO sensor with a short socket or connect the sensor to the sensor probe for storage. Otherwise, the sensor may have a shorter life or stable instructions may not be obtained.
- The short socket is used when storing. Do not throw it away.

Resetting the DO sensor when storing without having installed the short socket.

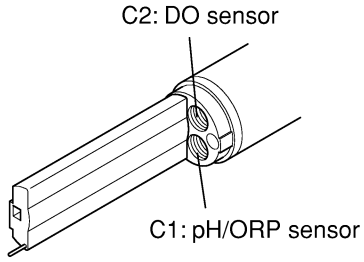
When leaving the DO sensor unattended for a brief period (1 or 2 days) without the short socket, the DO sensor can be reset by connecting it to the short socket or the probe. However, an amount of time corresponding to the period it was left unattended is necessary. If left unattended without being connected to the short socket or the probe for a long period (1 month), it cannot be reset.

Where to attach

1. The hole on the sensor probe in which each sensor is attached is determined by the type of sensor. Check the type of sensor and the assigned hole before attaching anything.

Important

- Installing the sensor in the wrong hole will damage both the sensor and sensor probe.

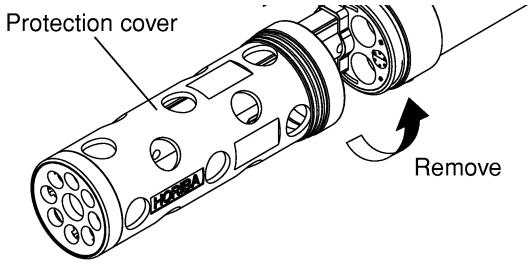


Installation procedure

Important

- With the U-22XD sensor probe install the DO sensor first and then the pH/ORP sensor.
- We recommend that the O-ring of the sensor be replaced with a new one each time the sensor is removed.

1. Remove the probe cap and remove the protection cover from the sensor probe.



When the protection cover's screws are firmly fixed in place and cannot be removed by hand, place a spanner on the protection cover and the surface of the cover guide and remove.

Do not try to remove the protection cover by hitting it or submitting it to shocks.

2. Apply silicon grease to the DO sensor's O-ring.

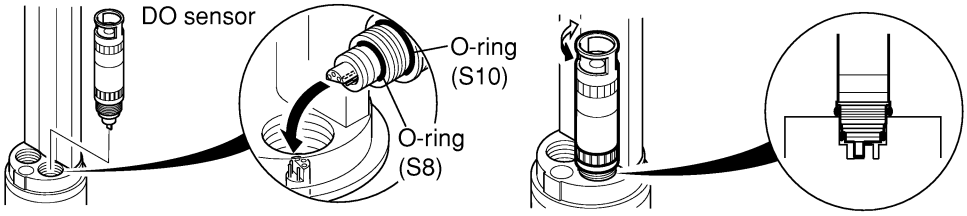
Make sure that no silicon grease gets on the connector.

3. Fit the DO sensor inside the sensor probe hole, being careful to align the shape of the connectors.

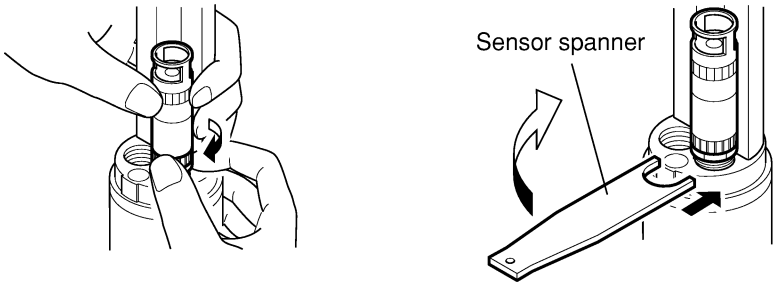
- Make sure that the O-ring is not scratched or twisted. Leakage will cause failures.
- Remove the DO sensor connected to the probe and, when reconnecting them, replace the O-ring (S8) on the smaller end of the DO sensor with a new O-ring.

Important

- Press the sensor slightly inward and try turning to check the fit. The sensor cannot be turned if inserted properly.



4. Turn the screw 2 or 3 turns by hand and then fully tighten with the sensor spanner.

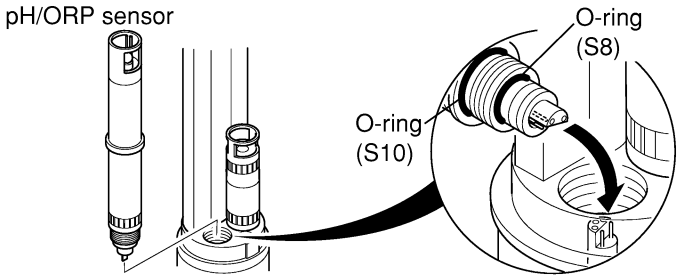


5. Apply silicon grease to the pH/ORP sensor's O-ring.

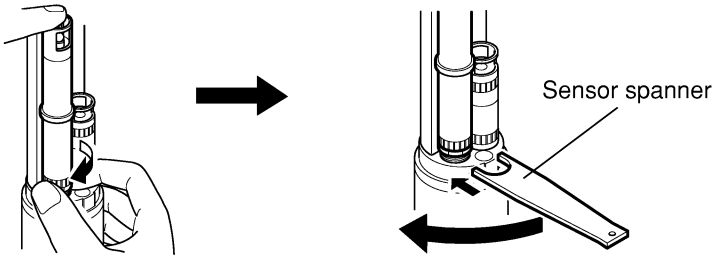
Make sure not to get silicon grease on the connector.

6. Fit the pH/ORP sensor inside the sensor probe hole, being careful to align the shape of connectors.

- Make sure that the O-ring is not scratched or twisted. Leakage will cause failures.
- Remove the pH/ORP sensor connected to the probe and, when reconnecting them, replace the O-ring (S8) on the smaller end of the pH/ORP sensor with a new O-ring.



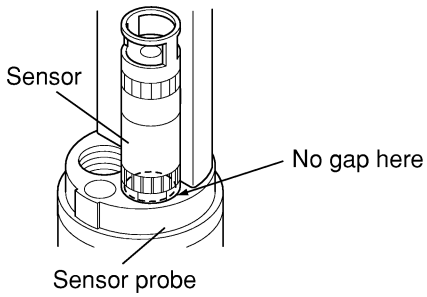
7. Holding the top of the pH/ORP sensor with your finger, turn the screw 2 or 3 turns by hand and then fully tighten with the sensor spanner.



⚠CAUTION

- Unless snugly attached, the sensor is not fully waterproof. The sensor is snugly fit inside the sensor probe when tightened as far as it will go.

Example for DO sensor



8. Attach the removed protection cover to the sensor probe as it was.

Important

- Before attaching each sensor to the sensor probe, do not soak the connector block in water.
- Be careful not to contaminate or wet the sensor probe or sensor connector.

Fasten the guard cover with your hand until it touches the end surface. If improperly fastened, it will slacken and, when storing the instrument, there will be a lack of humidity control. Fastening by hand is enough, do not use a spanner or other tool to fasten or the screws may break.

2.3.3 Installation and replacement of the battery

The U-20XD series is shipped from the factory with the battery packed separately.
When using the instrument for the first time or replacing the battery, perform the following procedure:

Type of battery:

Instrument (U-2000) ..... Alkaline battery 6LR61 (Manganese battery 6F22 [006P])
1 piece. (Battery for instrument operation)
Sensor probe ..... Alkaline batteries LR03 [AAA] (Manganese battery [R03])
3 pieces. (Battery for memory backup)

Notes on handling the battery

The improper use of battteries may cause leaks and explosion.
Observe the followings:

- Set the batteries in place properly while paying attention to the plus (+) and minus (-) poles.
- Do not use both an old and new batteries at a time or batteries of different types.
- Battteries for use in the instrument are not of the rechargeable type.
- Remove the batteries when not in use for a long.
  In case of leaks, wipe off the solution in the battery case thoroughly and place new battteries in position.

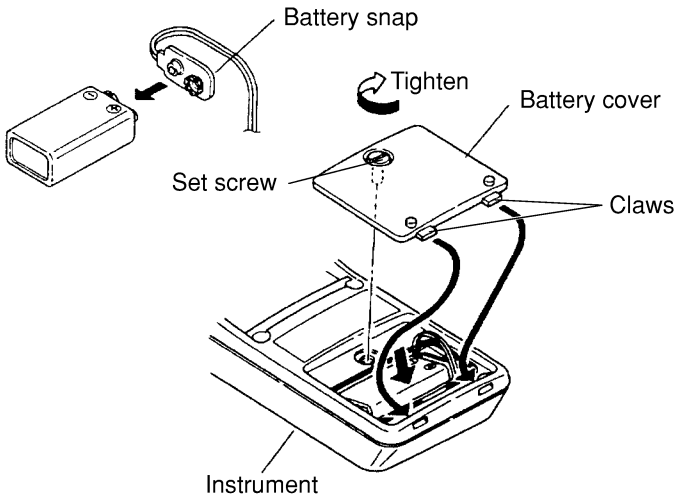
Note

- The battery originally attached to your unit is for monitor and the service life of the battery cannot be guaranteed.

Instrument (U-2000)

1. Loosen the set-screw on the battery cover and remove the cover.
2. Remove any old battery.
3. Fit the battery snaps to a new battery and insert the battery assembly into the instrument.
4. Insert the claws on the battery cover into the grooves in the instrument. Then tighten the set screw.

   The battery snap may be loose for some batteries. In such a case use radio pliers and tighten the metal snap fittings.

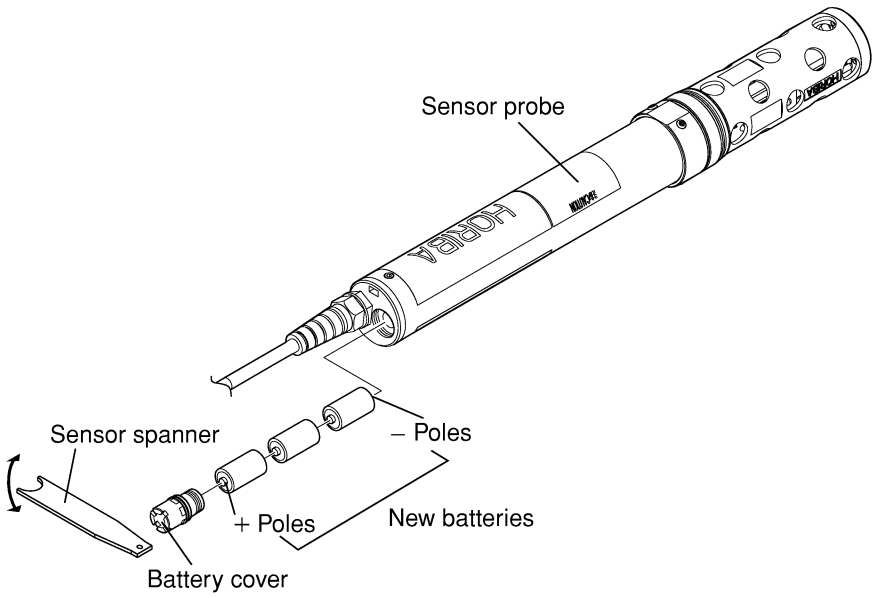


Important

- When removing the battery snap, do not pull it too strongly.

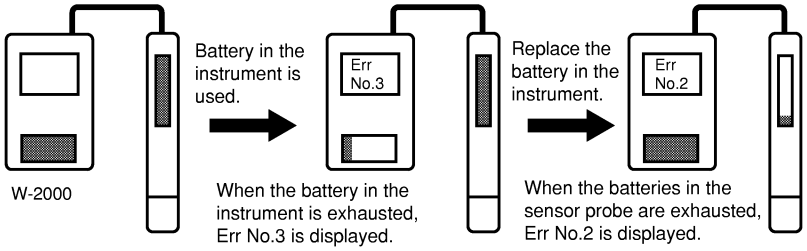
Sensor probe (for memory back up)

1. Remove the battery cover using a sensor spanner or a suitable object.
2. Remove any old batteries.
3. Insert new batteries making sure that the plus (+) and minus (-) poles match the terminals correctly.
4. To keep the sensor probe water-resistant, use a chip spanner as illustrated below and tighten the battery cover until the cover does not turn any more.



⚠ CAUTION

- When replacing the batteries of the sensor probe, be sure to connect the sensor probe to the instrument. Otherwise, the memory will be reset and all the data saved in the memory will disappear.
- When the sensor probe is connected to the instrument, battery in the instrument is consumed.

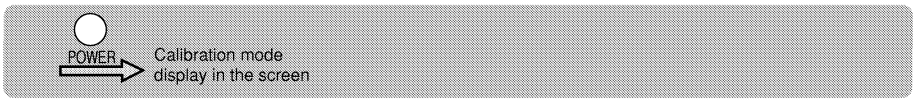


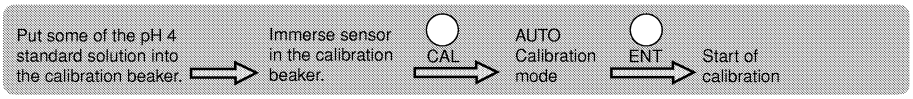
Note

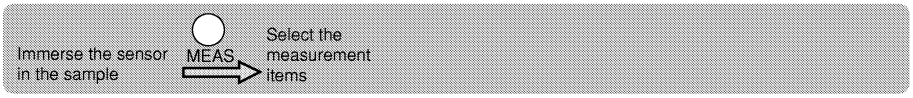
- The battery on the main unit is used up first allowing up to 30 hours use at room temperature. (When using alkaline batteries.)
- Life is reduced by approximately one half when manganese batteries are used.

3. Basic operation

The pH, conductivity (COND), turbidity (TURB), dissolved-oxygen (DO) and water depth (DEP) sensors can be calibrated automatically. Upon completion of this chapter, even beginners should be able to make measurements easily.





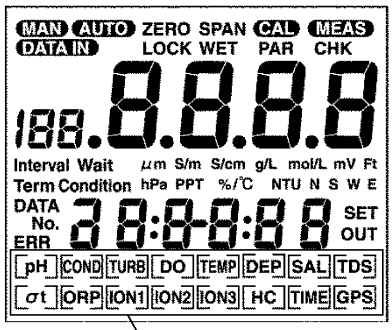


3.1 Key operations and mode switching

Measuring items and displays which are switched with the MEAS key

The items measurable with individual models are displayed. The items selected with the MEAS key will be indicated with [ ].

Example: In the pH Measurement mode: [pH]



Display block

The symbols displayed and their meanings are as follows:

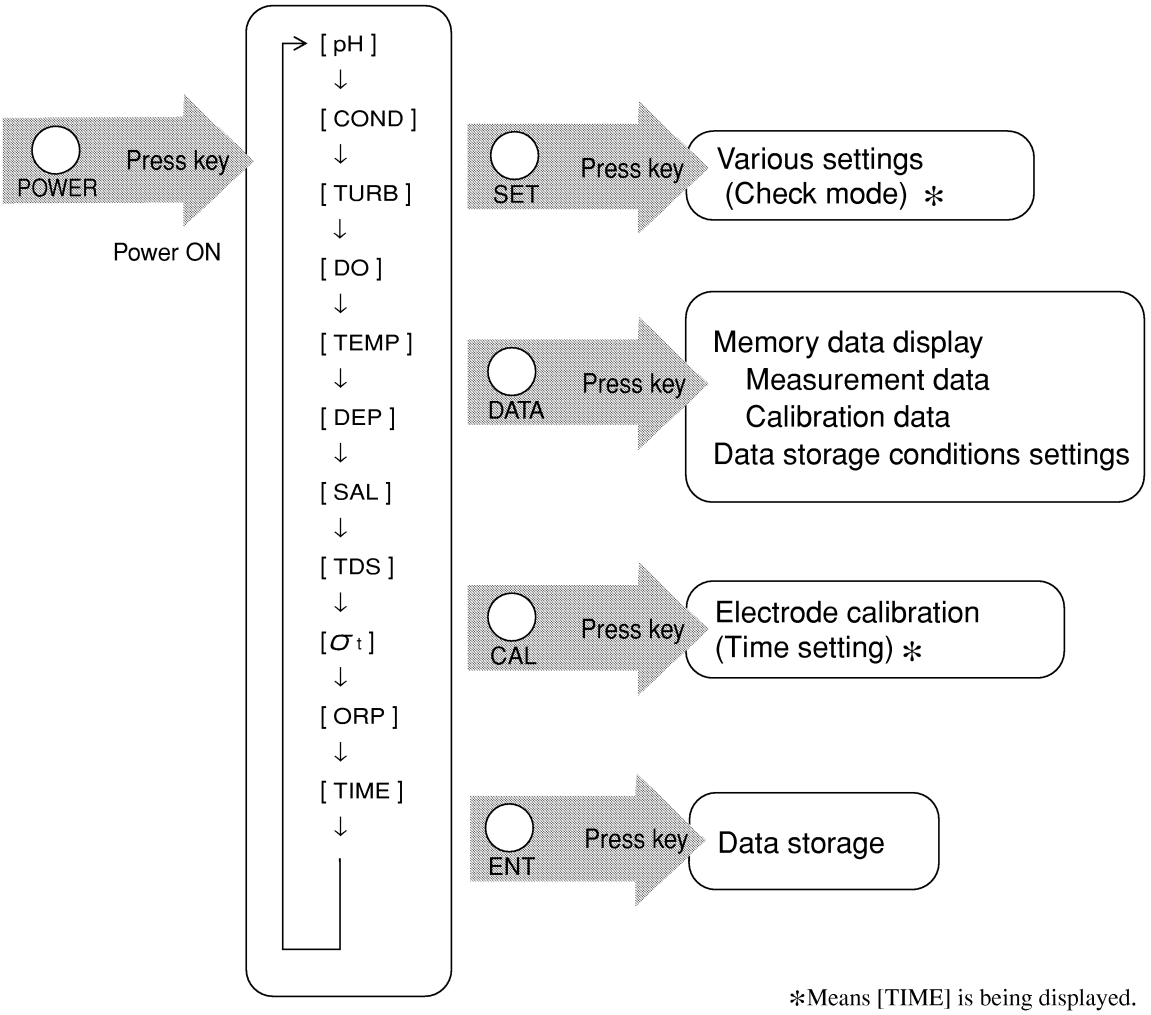
pH ..... pH
COND ..... Conductivity
TURB ..... Turbidity
DO ..... Dissolved-Oxygen
TEMP ..... Temperature
DEP ..... Depth
SAL ..... Salinity
TDS ..... Total dissolved solids
σ_t ..... Specific gravity of seawater
ORP ..... Oxidation-reduction potential
TIME ..... Display of date and time
GPS ..... G.P.S. (Global Positioning System) for imformation of position

Note

- [GPS] lights up when the optional G.P.S. sensor has been connected to the instrument and position information is received from the G.P.S. sensor during the measurement. For more information, refer to the instruction manual for the expansion units.

Measurement mode

MEAS When the MEAS key is pressed, the next measurement item appears.



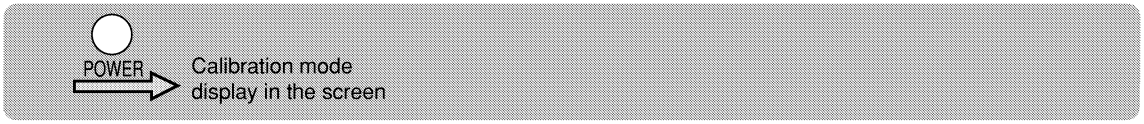
∗Means [TIME] is being displayed.

Note

- "*Measurement item setting*" on page 76 explains how to set the display so items are not displayed.

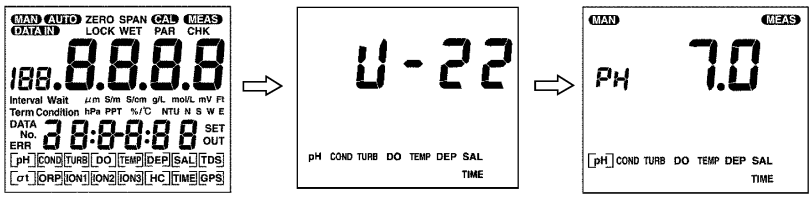
3.2 Operation procedure

3.2.1 Power ON

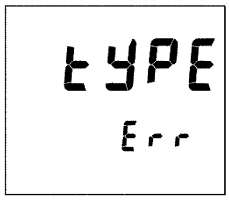


1. Press the **POWER** key.

The display will change in the order of All segment display → Sensor detector display → pH Measurement mode.



With the sensor probe is not connected,



is displayed.

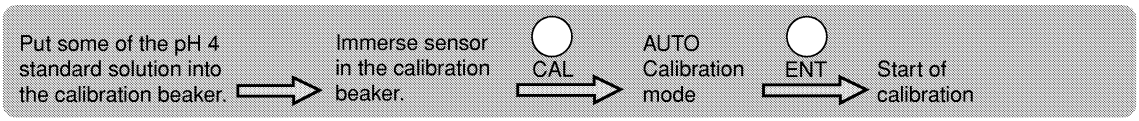
Before turning ON the instrument, connect the sensor probe properly.

3.2.2 AUTO calibration method

To obtain correct measurement, it is necessary to calibrate the sensor using the standard solution before performing measurement.
Previous calibration records shown in calibration log.
(☞ 4.3.2 *Calling up The calibration log*, page 43.)

Note

- In the AUTO calibration mode, the pH, COND, and TURB sensors are calibrated in the pH 4 standard solution, and the DO and DEP sensors in the atmosphere simultaneously.
  Calibrate contents at 25°C are as follows:
  pH: set at 4.01 (zero calibration) and the Span is the adjustment value at the factory when shipping.
  COND: 0.449 S/m (Span calibration), the Zero is the adjustment value at the factory when shipping.
  TURB: 0 NTU (zero calibration), the Span is the adjustment value at the factory when shipping.
  DO: 8.52 mg/L (Span calibration), the Zero is the adjustment value at the factory when shipping.
  DEP: 0 m (Zero calibration), the Span is the adjustment value at the factory when shipping.
- Values may be unstable if there is temperature fluctuation. Calibrate after waiting for about an hour.

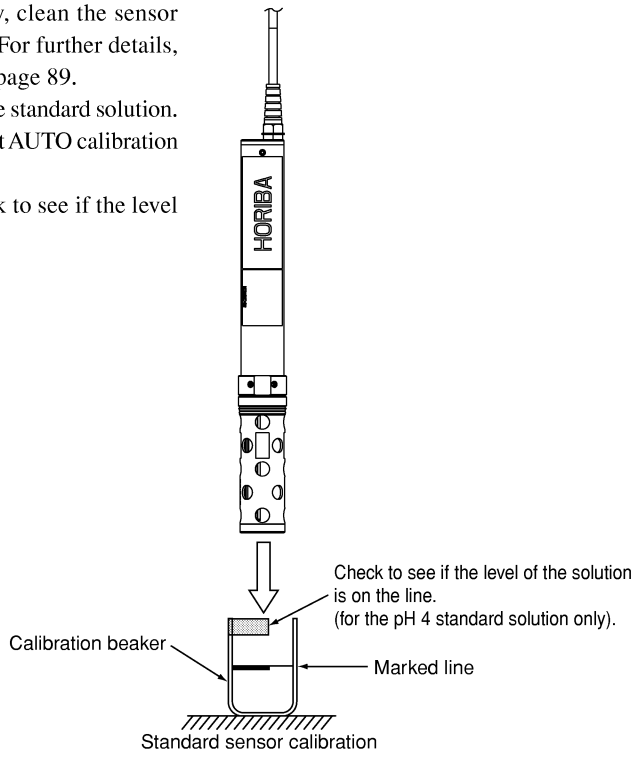


Calibrate using the following procedure.

1. Wash the sensor in distilled water a few times and put some of the pH 4 standard solution into the calibration beaker to the marked line. Then immerse the sensor in it.

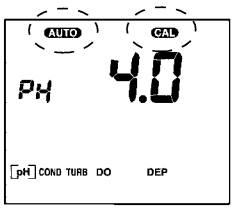
Important

- To carry out calibration for turbidity accurately, clean the sensor surface that will be soaked in standard solution. For further details, see "Troubleshooting for the TURB sensor" on page 89.
- Use the "100-4" manufactured by HORIBA for the standard solution. With other standard solutions, you cannot carry out AUTO calibration correctly.
- Use the label on the calibration beaker and check to see if the level of the calibration solution is on the label line.



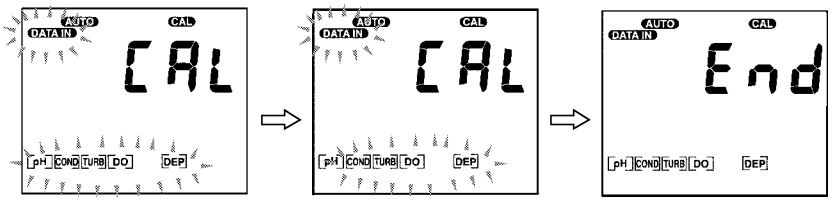
2. Press the **CAL** key in one of the Measurement modes pH, COND, TURB, DO and DEP.

AUTO and **CAL** appear and the instrument enters the AUTO Calibration mode.



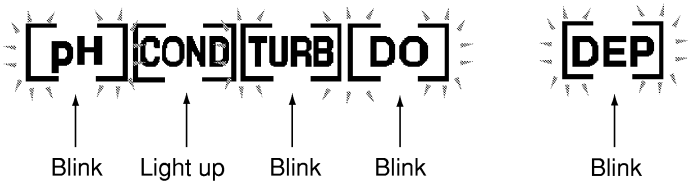
3. Press the **ENT** key to start AUTO Calibration.

Upon completion of all of the pH, COND, TURB, DO, and DEP modes, End will be displayed.
During calibration, **DATA IN** and [ ] for the selected measurement item blink. [ ] light up for the item of which calibration is finished.



With DATA IN is blinking
To stop calibrating the sensor .... press the CAL key.
To establish the calibration ..... press the ENT key.

Example: When COND calibration is finished:
[ ] for [COND] stops blinking and light up steadily.



Note

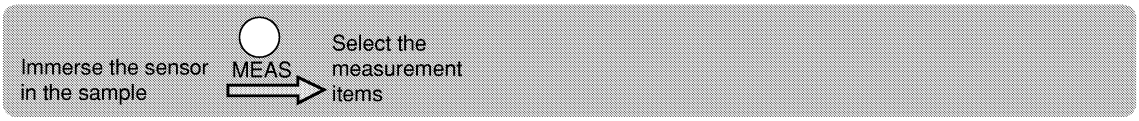
- [ ] continues to blink because calibration is not performed for the item for which an error has happened. If two or more errors happen, an error with a smaller number appears. (See pages 85 to 88 for these errors and ways to solve them.) These calibration errors disappear when the sensor is calibrated properly again, or when the instrument is turned ON again.
- Calibration should be performed for maximum three minutes. When the indications become stable, calibration should be finished.

4. Press the **MEAS** key to return to the Measurement mode.

Important

- Neutralize any basic pH 4 fluids before disposal.

3.2.3 Measurement



1. Immerse the sensor in the sample.

2. Select the measurement item.

Use the MEAS key to switch measurement items in the following order:

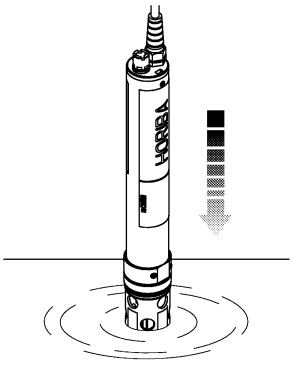
pH → COND → TURB → DO → TEMP → DEP → SAL → TDS → σ_t → ORP → TIME → then back to pH.

Note

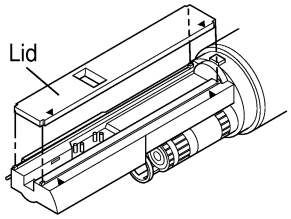
- [GPS] lights up when the optional G.P.S. sensor is connected to the instrument and position information is received from the G.P.S. sensor.
- The above measurement items can be changed by setting ""*Measurement item setting*" described on page 76.

Important

- When immersing the sensor probe in the sample, slowly lower the sensor probe into the sample.
- Dropping it from a height of 1m or more may cause damage to the sensor.



- Don't remove the COND/TURB lid during calibration or measurement.
- Attach the lid to the cell with fitting four corners and facing ▲ marks each other.



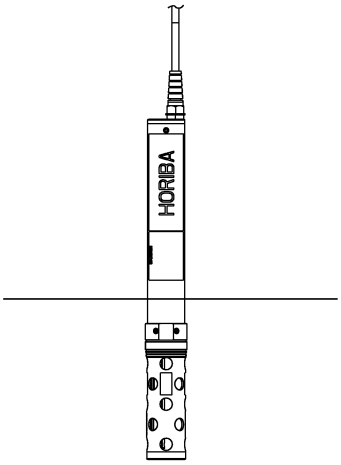
- Perform AUTO calibration after attaching the lid again, when the lid has been removed for the cleaning. A slight difference of the fitting position of the lid causes the difference of the indicated value for turbidity.
- Contacting with a different kind of metal, protection cover of the sensor probe may cause an error in measurement. Be careful not to let protection cover touch with any metal in measurement.

Two useful uses of the U-20XD Series models

Making measurements

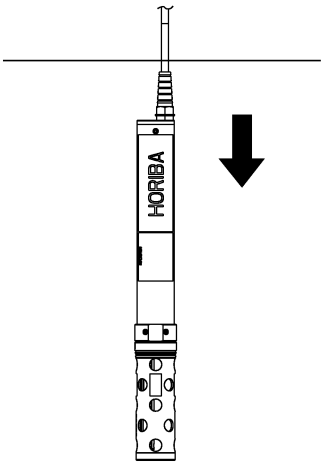
1. Manually storing the measurement data after checking the indication becomes stable

Example: After switching measurement items with the MEAS key, you can then store the measurement data after checking the indication becomes stable.
(☞ 4.1 *Manual storage of data while monitoring the measurement data*, page 34.)



2. Storing data

Example: Data can be stored continuously at constant intervals from the start of the automatic data storage. This function is useful in obtaining data in depth direction and in storing data continuously.
(☞ 4.2 *Automatic data storage*, page 36.)



Notes in obtaining data on depth

- When the instrument is placed at a depth of 100 m or more, the instrument may be broken.

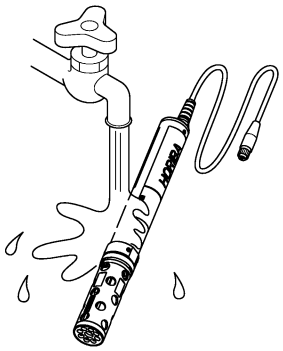
Notes for reliable measurements

- Any sensor contamination may affect measurements. Use the AUTO calibration mode to check for contamination on sensors about once a week for measurements.

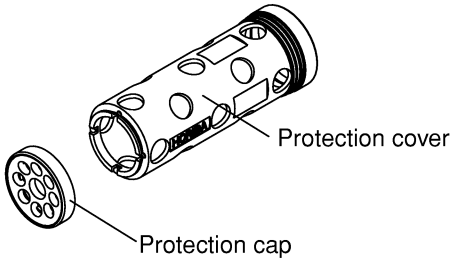
3.2.4 After completion of measurement

1. Turn the power to the instrument off.

2. Use tap water to completely wash off the sample on the sensor and then wipe waterdrops.



Remove the protection cover once and completely wash out with tap water the left over sample on the screws. Reinstall the cover after having wiped off the drops of water. If there is any sample (especially sea water) left over on the screws, rust may form which may prevent the protection cover from being removed. (☞ Installation procedure, page 18.)
Depending on the level of contamination, remove the rubber protection cap from the tip of the protection cover and wash out with tap water. Reinstall it after wiping off the drops of water.



3. Pour about 20 mL (about 2 cm from the bottom)of pure water in the probe cap and install it on the sensor probe. Place the rubber cap on the connector and store the instrument in the carrying case. (☞ 2.2.2 Sensor probe names, page 10.)

When storing with the ph/ORP and DO sensors attached to the probe, make sure to install the probe cap after having poured pure water into it.
Letting the ph/ORP and DO sensors get dry may cause deterioration of the instrument's performance. Should the sponge inside the probe cap be contaminated, replace it with a clean sponge (included).

Now you have read the description for performing measurements. For further information on how to use the instrument, refer to the chapters hereafter.

MEMO

4. Using the data memory function

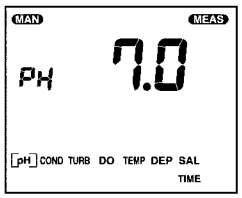
The data memory function can be used to store manualy measurement values with associated data numbers and to store automatically measurement values at fixed intervals (data logger).

4.1 Manual storage of data while monitoring the measurement data

Make sure MAN is displayed on the measurement screen. → ENT → Start data storage.

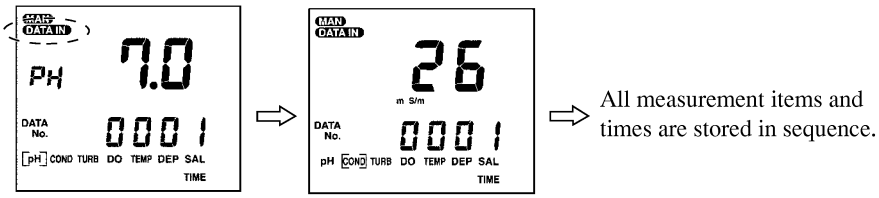
1. Make sure that **MAN** is displayed on the Measurement mode.

If **AUTO** is displayed, switch to **MAN** display.
(☞ page 35, Switch to **MAN** display on the measurement mode)



2. Press the **ENT** key.

Data storage starts, **DATA IN** and the data No. are displayed on the screen, and the measured value to be stored and the measurement item are displayed in order at approximately 0.5 second intervals.

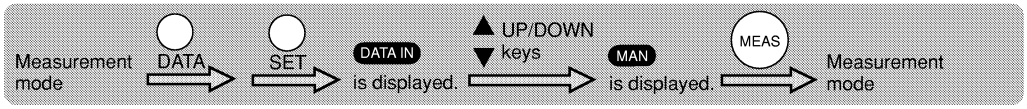


After the data is stored in memory, the screen returns to the original Measurement mode.

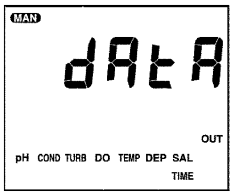
Note

- Up to 2880 sets of data can be stored in the memory.
  When 2880 sets of data have been stored in the memory, ERR 9 appears and no more data can be stored. In this case, "*Data memory clear*" while referring to page 78, and you can store new data in the memory.

When AUTO is displayed
Switch to MAN display on the measurement mode

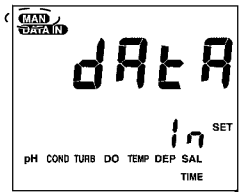


1. Press the **DATA** key in the Measurement mode.



2. Press the **SET** key.
DATA IN is displayed.

3. Press the **UP/DOWN** (▲ ▼) keys to display MAN.

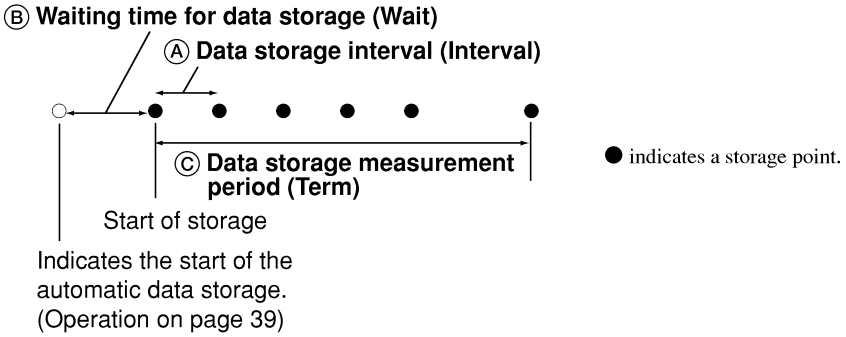


4. Press the **MEAS** key to return to the Measurement mode.

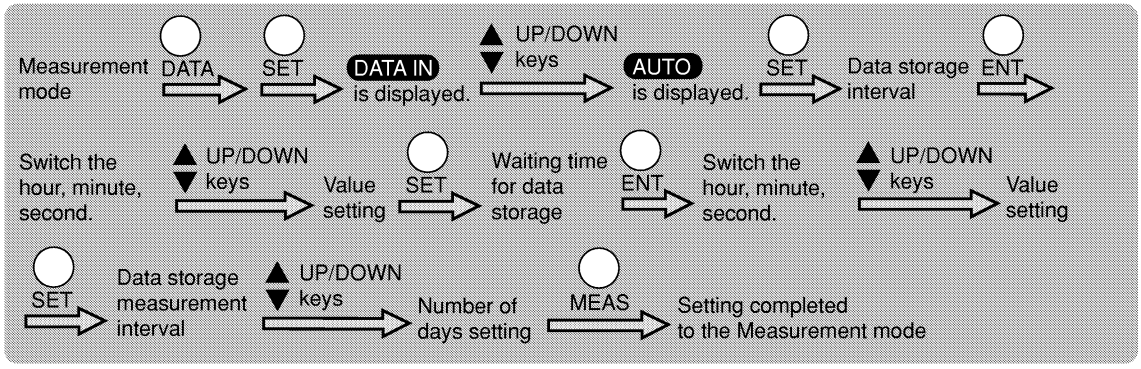
4.2 Automatic data storage

Measured values are stored automatically at constant time intervals. Before using the automatic storage, the following condition settings are required:

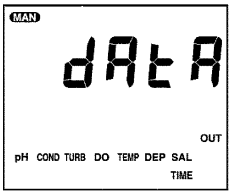
- Setting of data storage interval (4.2.1, step 4)
- Setting of waiting time for data storage (4.2.1, step 6)
- Setting of the data storage measurement period (4.2.1, step 8)



4.2.1 Data memory conditions settings



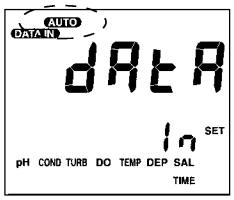
1. Press the **DATA** key in the Measurement mode.



2. Press the **SET** key.

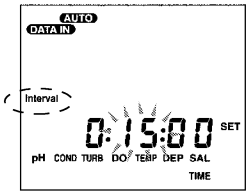
DATA IN is displayed.

3. Press the **UP/DOWN** (▲ ▼) keys to display **AUTO**.



4. Press the **SET** key to display the screen for setting the data storage interval Ⓐ.
"**Interval** " is displayed.

5. Press the **ENT** key to switch the among "hour", "minute" and "second" and set the value using the **UP/DOWN** (▲ ▼) keys.
(Data storage intervals can be set to 2 seconds to 24 hours.)
The current setting location will blink.

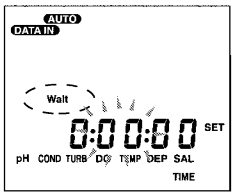


6. Press the **SET** key to display the screen for setting the waiting time for data storage Ⓑ.
"**Wait** " is displayed.

7. Press the **ENT** key to switch among "hour", "minute" and "second" and set the value using the **UP/DOWN** (▲ ▼) keys.
(The waiting time for data storage can be set to 2 seconds to 24 hours.)
The current setting location will blink.

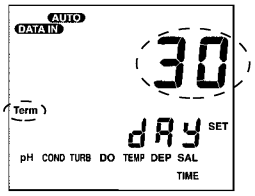
Important

- If wait time is set to "0", note that data is not stored in a memory the first time.



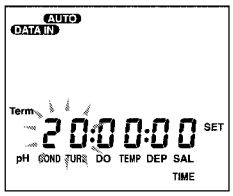
8. Press the **SET** key to display the screen for setting the data storage measurement period Ⓒ (number of days).
"**Term** " is displayed.

9. Use the **UP/DOWN** (▲ ▼) keys to set the value (number of days).



Setting of less than 24 hours

First set the number of days to 00 then press ENT key to select the "hour/minute/second" setting. Use the UP/DOWN (▲ ▼) keys to set the hour, the minute and second. During setting, the number to be set blinks.

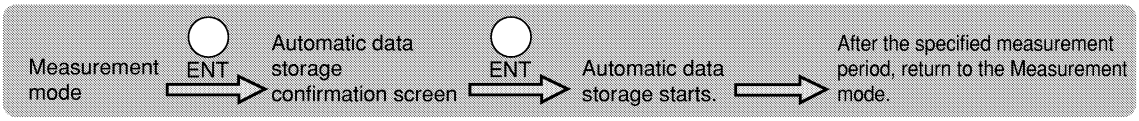


Note

- Press the SET key to return to step 4.

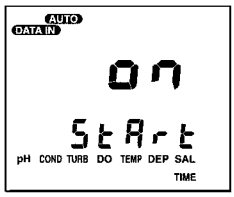
10. When the **MEAS** key is pressed, setting will be completed and the instrument will return to the Measurement mode.

4.2.2 Start of automatic data storage



1. Make sure that **AUTO** is displayed on the Measurement mode.

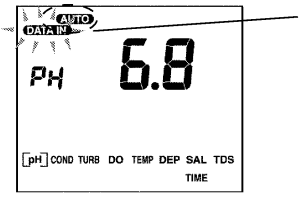
2. Press the **ENT** key. A confirmation screen will be displayed asking if you wish to start automatic data storage.



Note

- If you do not wish to proceed with automatic data storage, press the CAL key to return to the Masurement mode.

3. Press the **ENT** key to start automatic data storage.



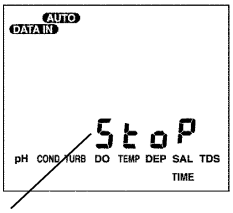
While **DATA IN** is blinking, the automatic data storage is being executed.

Note

- During the automatic data storage, measurement items can be switched by pressing the MEAS key.

Important

- During the automatic data storage, the ENT, SET, and DATA keys do not function and therefore calibration, setting change and stored data display cannot be performed.
- To stop automatic data storage, press the CAL key.



Confirmation display for canceling automatic data storage appears.
To stop the automatic data storage ..... Press the ENT key.
To return to the screen for the automatic data storage ... Press the DATA key.

4. After the specified measurement period, DATA IN disappears and the instrument returns to the normal Measurement mode.

Note

- When the instrument is turned on, AUTO lights up and DATA IN blink if automatic data storage is being performed with the sensor probe.

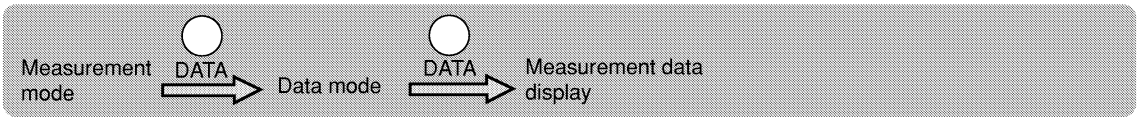
Notes for automatic data storage

- For long-term data storage, replace the sensor probe battery with a new one.
- You can remove the connector from the main unit. It can still be used for up to 60 hours at room temperature with the battery in the sensor probe (alkaline battery). Life is reduced by approximately one half when manganese batteries are used.
- If the sensor probe is connected to the instrument for monitoring, the instrument battery is first consumed to protect the memory of the sensor.
- When 2880 sets of data have been stored in the memory, ERR 9 appears and no more data can be stored. The automatic data storage is automatically ended and the instrument returns to the normal Measurement mode.

4.3 Calling up data from the memory

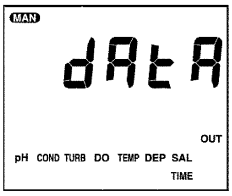
4.3.1 Calling up measurement data

Reading out data that has been stored manually or automatically.



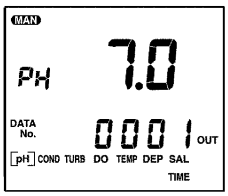
1. Press the **DATA** key in the Measurement mode.

The instrument goes to the DATA mode.



2. Press the **DATA** key.

The measurement data is displayed.
Data you want to call can be displayed by selecting a measurement item and data No.



DATA key ..... Selects switching of measurement item or memory data No.

When switching measurement items: Measurement item blinks.
When switching data No. : Data No. blinks.

UP/DOWN (▲▼) keys .... Switch measurement item or No. which has been selected with the DATA key.

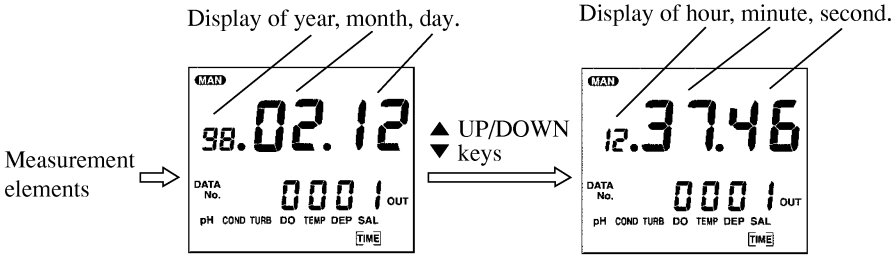
Note

- If you push the CAL key, only the data numbers will be displayed, allowing rapid changing of the numbers. Push the UP/DOWN (▲▼)keys to find the number, then press the SET key to display the data.

3. Press the **DATA** key.

TIME data

Use the UP/DOWN (▲▼) keys to switch between “Yer, Month, Day” and “Hour, Minute, Second”.

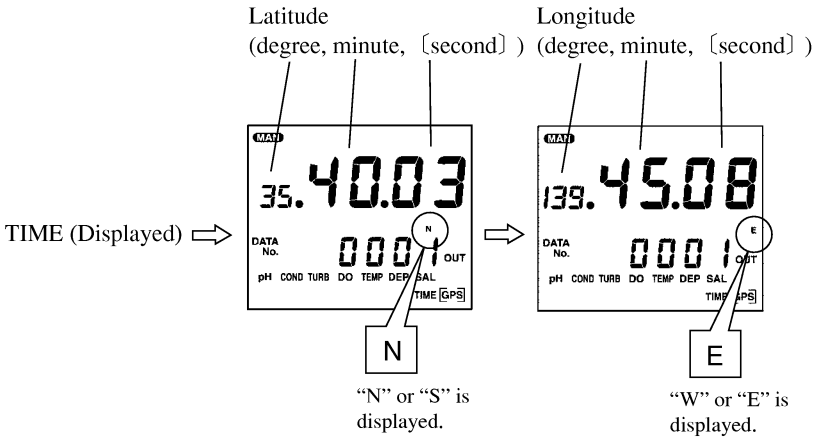


Note

- The time in the automatic memory can be out by about 2 seconds.

G.P.S. data (only when G.P.S. data is present)

Use the UP/DOWN (▲ ▼) keys to switch between “Latitude” and “Longitude”.



Latitude N → The North latitude S → The South latitude
Longitude E → The East longitude W → The West longitude

ENT key ..... Prints all measurement data for the displayed memory data item.
(when the printer is connected to the instrument)

Useful uses of keys in automatic storage

SET + UP (▲) key ..... Displays the first part of the next data automatically stored.
SET + DOWN (▼) key ..... Displays the first part of the previous data automatically stored.
If there is manual data, then the previous or next manual data is shown.

Display for automatic storage

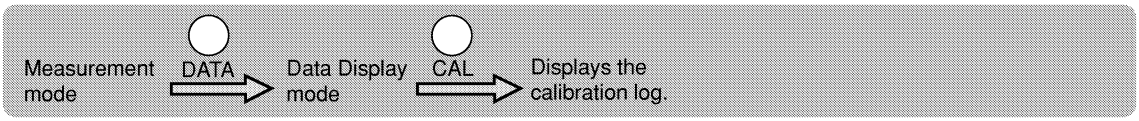
For the first and last data in one session of automatic storage the following identification marks are displayed in front of the values representing the data Nos.:
[: displayed for the first data in automatic storage.
] : displayed for the last data in automatic storage.

Note

- When the MEAS key is pressed, data calling is stopped and the instrument returns to the Measurement mode.
- Data is called from the sensor probe so to get one piece of data takes about one second.

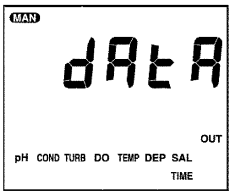
4.3.2 Calling up the calibration log

A calibration log is a record containing the “year, month, day” and “hour and minute” of the last calibration of individual measurement items and their calibration method. The instrument automatically stores the calibration log.



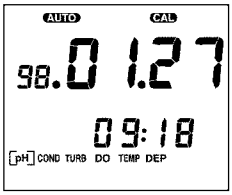
1. Press the **DATA** key in the Measurement mode.

The instrument goes to the DATA Display mode.



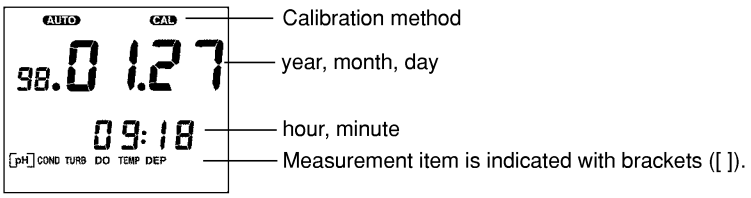
2. Press the **CAL** key.

The calibration log is displayed.



UP/DOWN (▲▼) keys: Switch the measurement item.
ENT key: Prints the entire calibration log. (when the printer is connected to the instrument)

Calibration log.



Calibration method

AUTO CAL	: AUTO calibration
MAN ZERO CAL	: Manual zero calibration
MAN SPAN CAL	: Manual span calibration
MAN ZERO SPAN CAL	: Manual zero calibration and span calibration

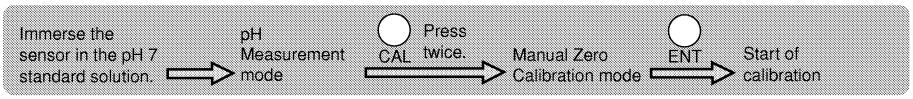
Note

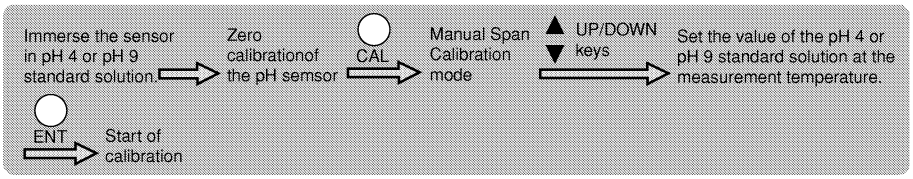
- Press the MEAS key to abort the data calling and return to the Measurement mode.

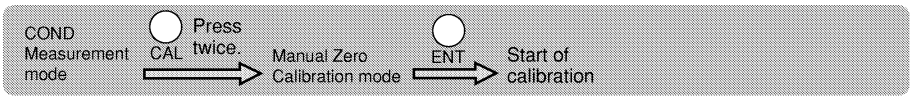
MEMO

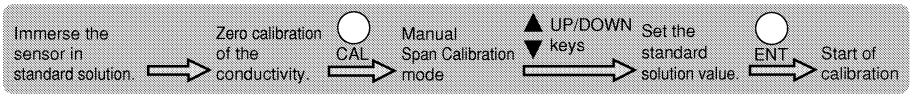
5. Techniques for more accurate measurement

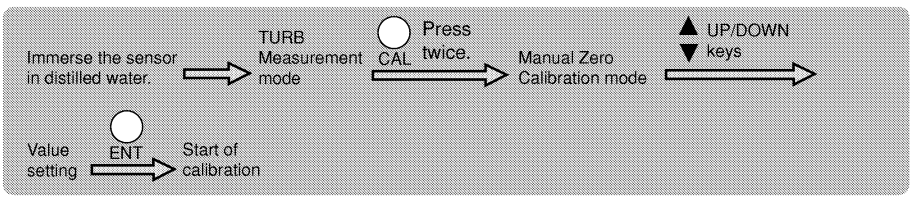
In normal operation, calibration using the AUTO Calibration mode described earlier in the basic operation section provides sufficient accuracy. However, for more accurate measurement, manual calibration is effective. When measurement with high-accuracy extended display is needed, be sure to perform manual calibration.
Attention: The extended display mode is entered automatically when manual calibration is selected.

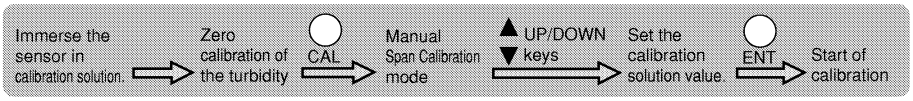


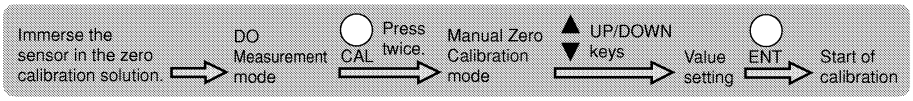


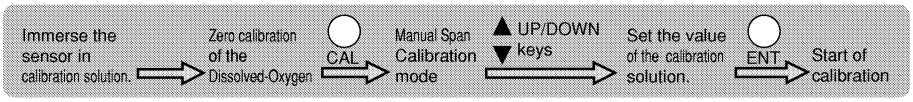


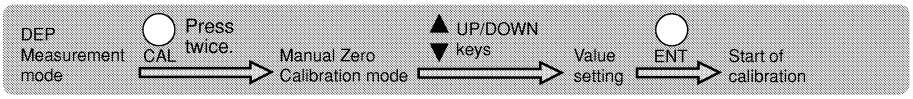


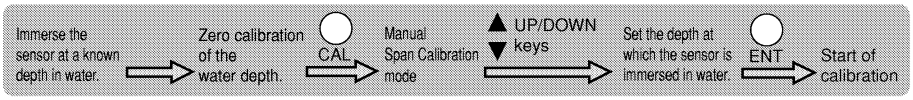


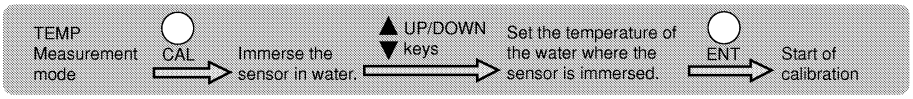






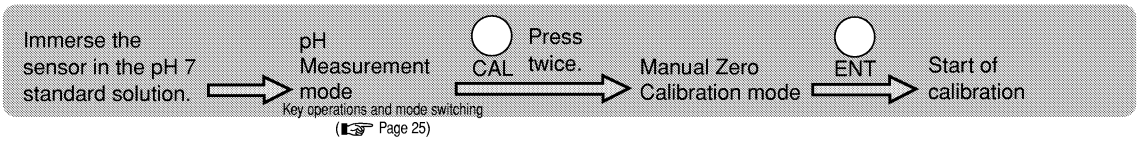






5.1 Manual pH calibration

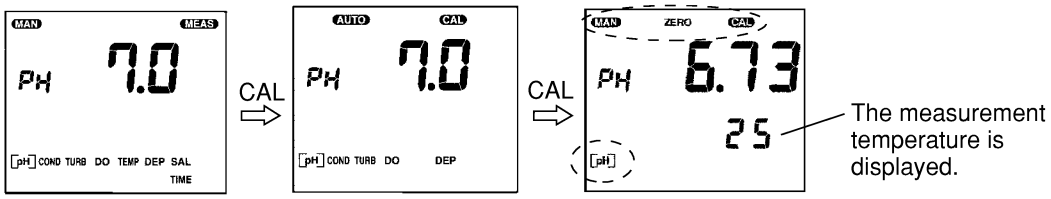
5.1.1 Zero calibration



1. Wash the sensor two or three times using distilled water, then pour some pH 7 standard solution into the calibration beaker, and immerse the sensor in it.

2. Press the **CAL** key twice in the pH Measurement mode.

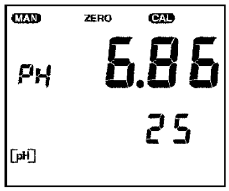
When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.



Manual Zero Calibration mode

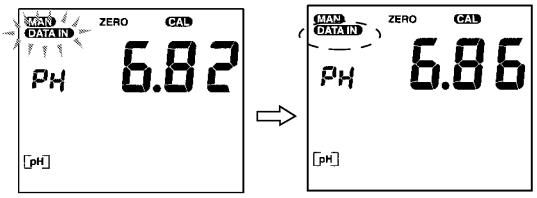
3. Use the **UP**/**DOWN** (▲ ▼) keys to input the value for the pH 7 standard solution at the measurement temperature.

(☞ *8. Reference data*, page 96.)



4. Press the **ENT** key.

The manual zero calibration starts.



End of calibration

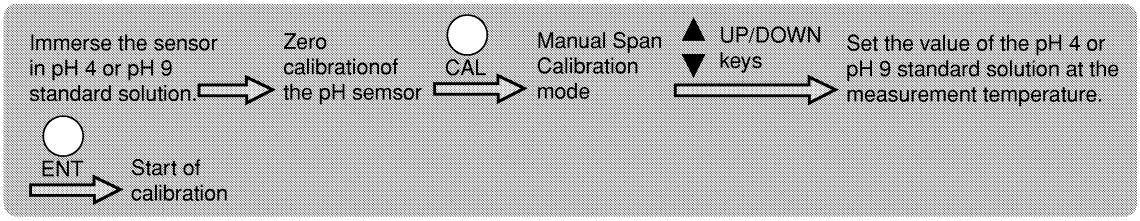
The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

To stop calibrating the sensor .... Press the CAL key.

To establish the calibration ..... Press the ENT key.

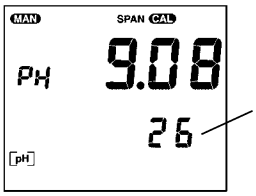
5.1.2 Span calibration



1. Wash the sensor two or three times using distilled water, then pour some pH 4 or pH 9 standard solution into the calibration beaker, and immerse the sensor in it.

2. After the zero calibration of the pH sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
MAN, **SPAN** and **CAL** light up.

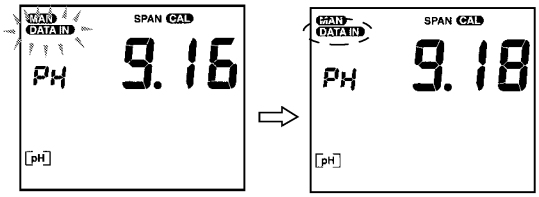
3. Use the **UP/DOWN** (▲ ▼) keys to set the value for the pH 4 or pH 9 standard solution at the measurement temperature.



The measurement temperature is displayed.

4. Press the **ENT** key.

The manual span calibration starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

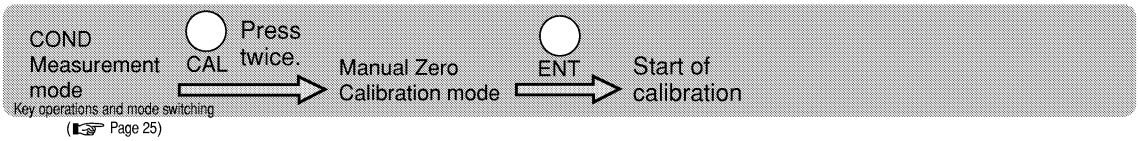
Note

- When the SET and CAL keys are pressed during the manual pH calibration mode, the calibration data for the pH sensor can be deleted.

5.2 Manual conductivity (COND) calibration

The U-20XD series models can measure conductivity (COND) in the range from 0.90 to 9.99 S/m. Depending on the concentration of the sample, these models automatically select the most suitable measuring range from three ranges: 0.0 to 99.9 mS/m, 0.090 to 0.999 S/m, and 0.90 to 9.99 S/m. The zero point is common to the three measuring ranges.

5.2.1 Zero calibration

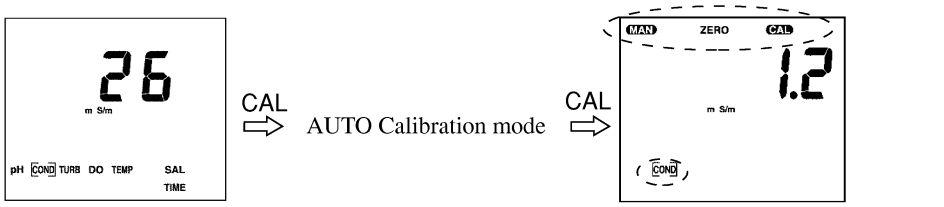


Key operations and mode switching
(☞ Page 25)

1. Wash the conductivity (COND) sensor two or three times using distilled water. Completely remove the water on the sensor and calibrate the instrument in the atmosphere.

2. Press the **CAL** key twice in the Conductivity (COND) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.

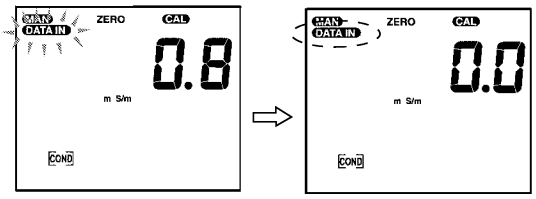


Manual Zero Calibration mode

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5.2.2 Span calibration

Preparation of calibration solution (Potassium chloride (KCl) standard solution)

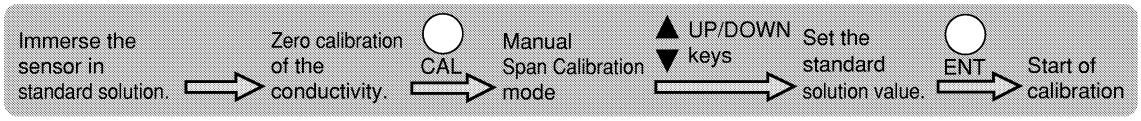
Dry Potassium chloride (KCl) powder (high-grade commercially available) at 105 °C for two hours, and leave it to cool in a desiccator.
Consult the following table and measure a portion of potassium chloride (KC1), then prepare standard potassium chloride (KC1) solution following the procedure below.

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	0.373	0.0 to 99.9 mS/m
0.050 mol/L	0.667 S/m	3.73	0.090 to 0.999 S/m
0.500 mol/L	5.87 S/m	37.2	0.90 to 9.99 S/m

1. Dissolve the weighed Potassium Chloride (KCl) in distilled water.
2. Put the dissolved Potassium Chloride (KCl) into a 1 L measuring flask, and fill to the 1 L mark with distilled water.

Calibration procedure

Perform the span calibration using the three types of standard solution as follows.

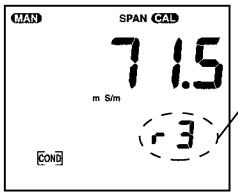


Important

- Set the temperature of the span standard solution to 25 ± 5 °C.
- The sensor should be calibrated in the three standard solutions in the order of increasing concentration.

1. Wash the sensor two or three times using distilled water, then pour some standard solution into the calibration beaker, and immerse the sensor in it.
2. After the zero calibration of the conductivity (COND) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
   MAN, **SPAN** and **CAL** light up.

3. Use the **UP/DOWN** (▲ ▼) keys to set the standard solution value.



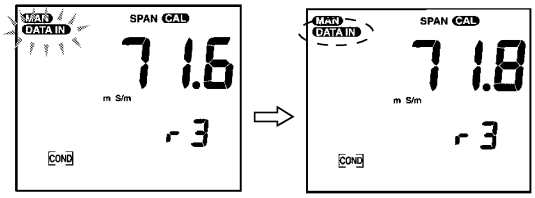
The calibration range is displayed.

Note

- The sensor automatically identifies the calibration solution and the relevant calibration range is displayed.
  - r 1 : 0.90 to 9.99 S/m
  - r 2 : 0.090 to 0.999 S/m
  - r 3 : 0.0 to 99.9 mS/m

4. Press the **ENT** key.

The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **CAL** key and use each standard solution and perform steps 1 to 4 above for calibration.

6. Press the **MEAS** key to return to the Measurement mode.

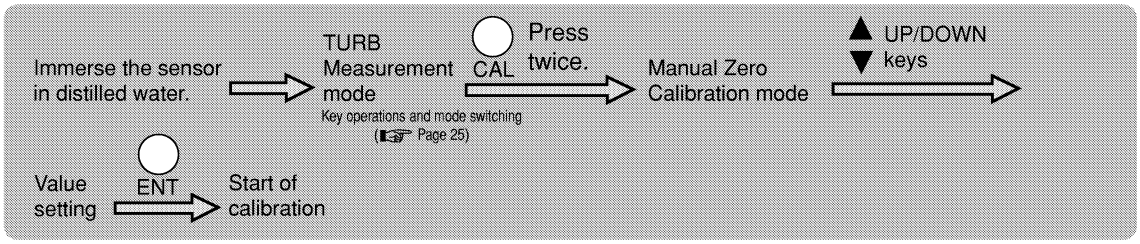
Note

- When the SET and CAL keys are pressed during the manual Conductivity (COND) Calibration mode, the calibration data for the conductivity (COND) sensor can be deleted.
- Perform the calibration again after deleting the present calibration data when calibration error occurs and the calibration cannot be performed.
- Perform the calibration again after deleting the present calibration data when the value cannot be read off because of unsettled digit of the measurement value.

5.3 Manual turbidity (TURB) calibration

5.3.1 Zero calibration

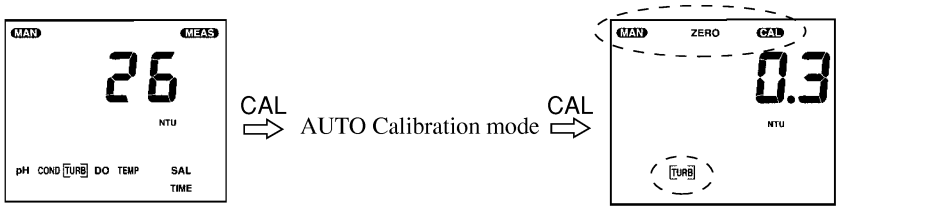
In zero calibration, distilled water is used as a calibration solution. If you cannot obtain distilled water, you may use ion exchange water, which can be considered to have a turbidity of zero.
When the turbidity (TURB) sensor is calibrated, it is particularly important that the probe is completely contamination-free. Do not use a contaminated probe. Otherwise unreliable calibration will result.



1. Wash the sensor two or three times using distilled water, then place some distilled water into the calibration beaker, and immerse the sensor in it.

2. Press the **CAL** key twice in the Turbidity (TURB) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.

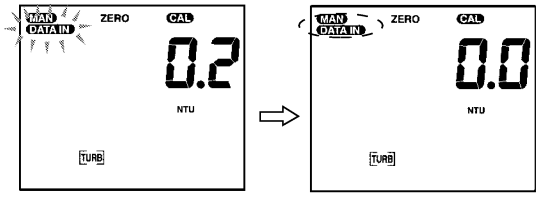


Manual Zero Calibration mode

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is started.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

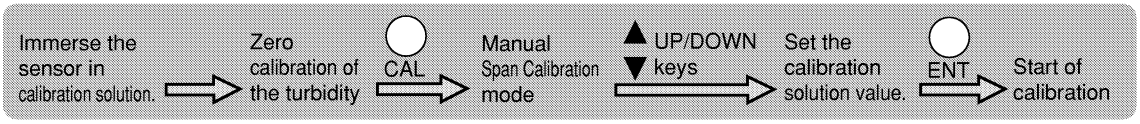
With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5.3.2 Span calibration

Preparation of calibration solution

Weigh out 5.0 g of hydrazine sulfate, and dissolve it in 400 mL of distilled water. Next dissolve 50 g of hexamethylene tetramine in 400 mL of distilled water, and mix the two solutions together. Finally add distilled water until the total solution volume is 1000 mL, and mix well. Store this solution at a temperature of 25 ± 3 °C for 48 hours. The turbidity value (TURB) of this solution is equivalent to 4000 NTU.
Use the solution as span calibration solution for turbidity (TURB) of 800 NTU by diluting this solution by a factor of 5 (use a pipette to measure 50 mL of the 4000 NTU solution and pour it into a 250 mL measuring flask, and add 200 mL of distilled water).

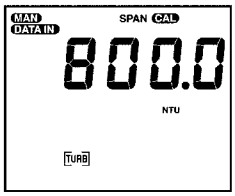
Calibration procedure



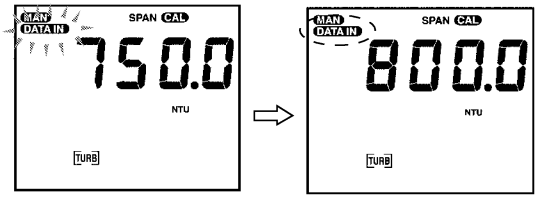
1. Wash the sensor two or three times using distilled water, then pour standard solution into a calibration beaker, and immerse the sensor in it.

2. After the zero calibration of the turbidity (TURB) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
MAN, **SPAN** and **CAL** light up.

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 800.0.



4. Press the **ENT** key.
The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Important

- When it is known beforehand that the solution for measurement has a low turbidity (0 to 100 NTU), calibrate the sensor in the span calibration solution of 80 NTU. To prepare an 80 NTU calibration solution, dilute the 4,000 NTU calibration solution with distilled water by a factor of 50.

Note

- When the SET and CAL keys are pressed during the manual Turbidity (TURB) Calibration mode, the calibration data for the turbidity (TURB) sensor can be deleted.

5.4 Manual Dissolved-Oxygen (DO) calibration

It is necessary to prepare a new calibration solution each time directly before calibration of the Dissolved-Oxygen (DO) sensor.

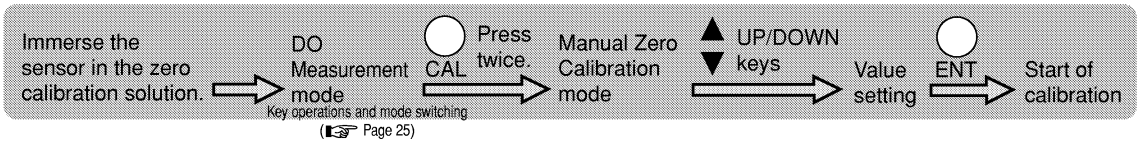
5.4.1 Zero calibration

Use ion exchange water or tap water with sodium sulfite dissolved in it.

Preparation of calibration solution

Add approximately 50 g of sodium sulfite to 1,000 mL of water (either ion exchange water or tap water) and stir the mixture to dissolve the sodium sulfite in it.
The calibration beaker (included) cannot be used to manually calibrate the DO sensor. Use a container that can immerse the DO sensor.

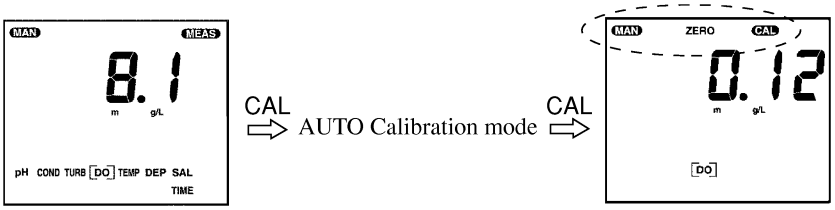
Calibration procedure



1. Wash the sensors 2 to 3 times with pure water and immerse the DO sensor completely in zero calibrated liquid.

2. Press the **CAL** key twice in the Dissolved-Oxygen (DO) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.

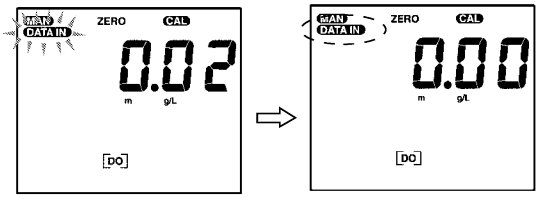


Manual Zero Caliburation mode

3. After the display has stabilized, use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

Important

- After calibration, use tap water to clean the sensor.

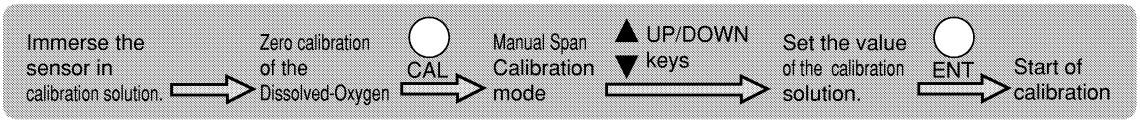
5.4.2 Span calibration

Use ion exchange water or tap water with saturated dissolved oxygen as the span calibration liquid.

Preparation of standard solution for span calibration

Pour 1 to 2 liters of water into a container (either ion exchange water or tap water).
Using a pneumatic pump, feed air into the water and froth up the solution until oxygen is saturated.
The calibration beaker (included) cannot be used to manually calibrate the DO sensor.
Use a container that can immerse the DO sensor.

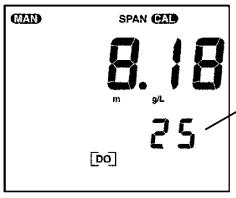
Calibration procedure



1. Wash the sensors 2 to 3 times with pure water and immerse the DO sensor completely in span calibrated liquid.

2. After the zero calibration of the Dissolved-Oxygen (DO) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
MAN, **SPAN** and **CAL** light up.

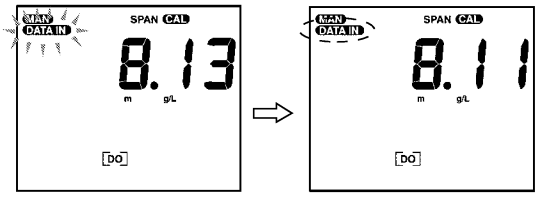
3. After the display has stabilized, use the **UP**/**DOWN** (▲ ▼) keys to set the amount of saturated dissolved oxygen in water at the temperature.



The temperature setting is displayed.
Refer to the table given on page 56 and set a value equivalent to the amount of saturated dissolved oxygen at the temperature.

4. Press the **ENT** key.

The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Note

- When the SET and CAL keys are pressed during the manual Dissolved-Oxygen (DO) calibration mode, the calibration data for the dissolved-oxygen (DO) sensor can be deleted.

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

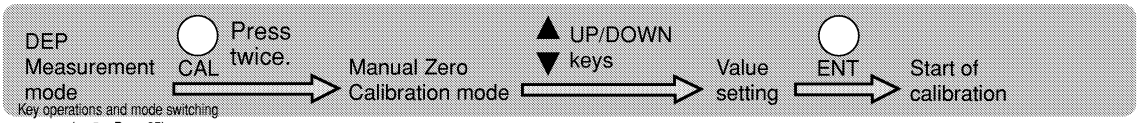
ISO5814

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	12.45	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

AUTO calibration is based on the JIS tables. When you need the measured data based on ISO, calibration should be done according to the procedure of span calibration.

5.5 Water depth (DEP) calibration

5.5.1 Zero calibration

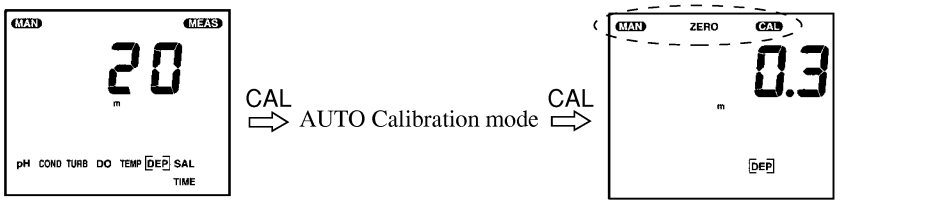


Key operations and mode switching (☞ Page 25)

1. Immerse the sensor in the sample water for approximately 30 minutes so that sensor probe and sample temperatures become the same.

2. Press the **CAL** key twice in the Water Depth (DEP) Measurement mode.

When the instrument enters the Manual Zero Calibration mode, **MAN**, **ZERO** and **CAL** light up.



Manual Zero Calibration mode

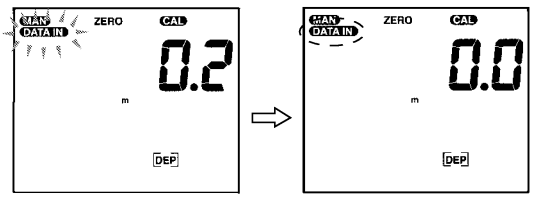
Important

- Sensor probe should be immersed to the depth where the battery cover comes level with the surface. And the level is uses as 0 m in depth.

3. Use the **UP/DOWN** (▲ ▼) keys to set the value to 0.0.

4. Press the **ENT** key.

The manual zero calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

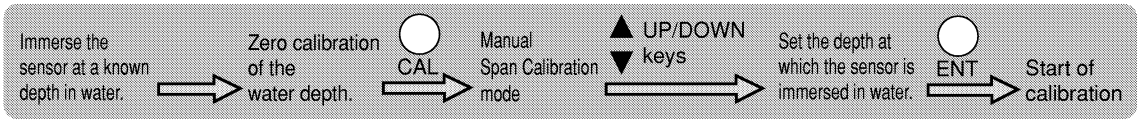
To stop calibrating the sensor .... Press the CAL key.

To establish the calibration ..... Press the ENT key.

Important

- Since the water depth (DEP) sensor depends greatly on temperature, calibrate the sensor at the same temperature as the sample for more accurate measurement.
- Use the AUTO Calibration mode because calibration error becomes large when using in a place with flow velocity or where it is shallow.

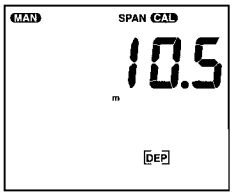
5.5.2 Span calibration



1. Immerse the sensor at a known depth in water. (Set the depth of the lid for memory backup battery as the depth setting.)

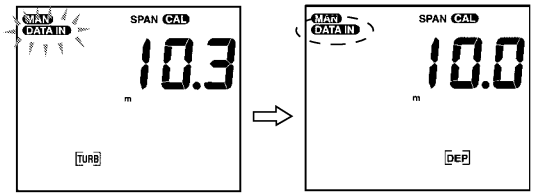
2. After the zero calibration of the water depth (DEP) sensor, press the **CAL** key to make sure that the instrument is in the Manual Span Calibration mode.
MAN, **SPAN** and **CAL** light up.

3. Use the **UP/DOWN** (▲ ▼) keys to set the depth at which the sensor is immersed in water.



4. Press the **ENT** key.

The manual span calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

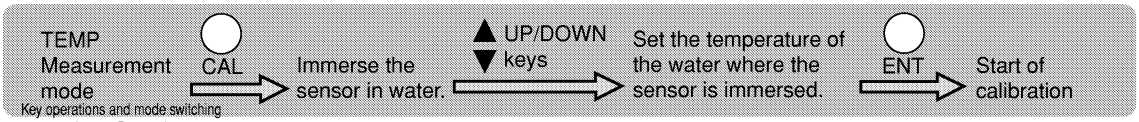
With DATA IN is blinking
To stop calibrating the sensor .... Press the CAL key.
To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Note

- When the SET and CAL keys are pressed during the manual Water depth (DEP) Calibration mode, the calibration data for the water depth (DEP) sensor can be deleted.

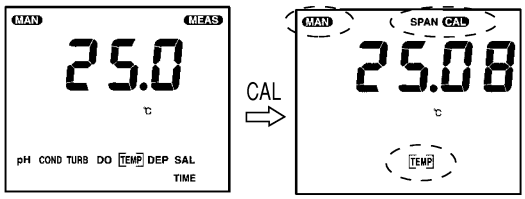
5.6 Temperature (TEMP) calibration



Key operations and mode switching
(☞ Page 25)

1. Press the **CAL** key in the Temperature (TEMP) Measurement mode.

Select the manual calibration mode.

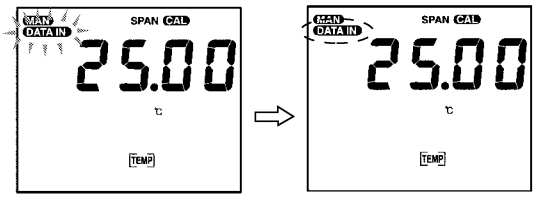


2. Immerse the sensor in water at a known temperature.

3. Use the **UP/DOWN** (▲ ▼) keys to set the temperature of the water where the sensor is immersed as a calibration value.

4. Press the **ENT** key.

The manual calibration is starts.



End of calibration

The measured value is displayed during calibration, and **DATA IN** blinks until the indicated value stabilizes. When the indicated value has stabilized, **DATA IN** lights up and the calibration finishes.

With DATA IN is blinking

To stop calibrating the sensor .... Press the CAL key.

To establish the calibration ..... Press the ENT key.

5. Press the **MEAS** key to return to the Measurement mode.

Note

- When the SET and CAL keys are pressed during the manual Temperature (TEMP) calibration mode, the calibration data for the temperature (TEMP) sensor can be deleted.

5.7 Operation check using ORP standard solution

Note

- Standard solution is not used only for calibration of the meter, but to confirm whether or not the condition of electrodes is good.

1. Add 250 mL pure (ion exchange) water to one packet of any of the below listed standard solutions and mix well.
   When mixing, the excess quinhydrone (a black powder) will float to the surface of the solution.
2. Immerse a washed and dried ORP electrode in the ORP standard solution and measure the mV value.
3. If the electrode and the meter, itself, are working correctly, numerical values within 15 mV or less of those listed in Table 1 should be obtained.
4. If measurements that fall within 15 mV of the values listed above are not obtained using this method, measure the solution again after replacing the reference electrode internal solution and removing the dirt from the surface of the metal electrode by moistening a cotton swab with alcohol or a neutral cleaning agent and lightly rubbing the electrode or by soaking the electrode in diluted nitric acid (1:1 nitric acid).
5. If measurements within 15 mV of the values listed above are still not obtained after re-measuring, the reference electrode or the meter may be faulty. Either replace the electrode or have the meter inspected.

Important

- If the prepared ORP standard solution is allowed to stand in open air for one hour or more, it may undergo transformation. For this reasons ORP standard solution that has finished being prepared cannot be stored.
- When measuring a solution that has 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, use the meter after immersing the electrodes in the solution again and mixing it thoroughly.

Precautions when measuring actual samples

- Note that when measuring the ORP of solution that has 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 water is allowed to stand, its ORP undergoes big changes. Always measure alkaline ion water promptly.

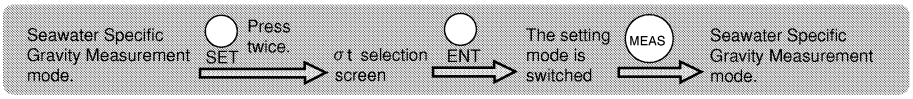
ORP standard solution

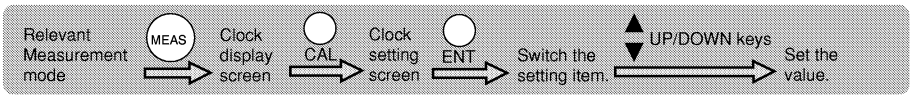
There are two kinds of standards substances. Under normal circumstances, it is sufficient to use only the one type of substance that is closest to the measured value.

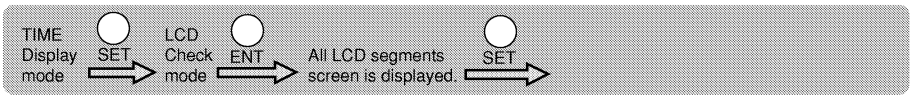
Indicated value of ORP standard solution at various temperatures

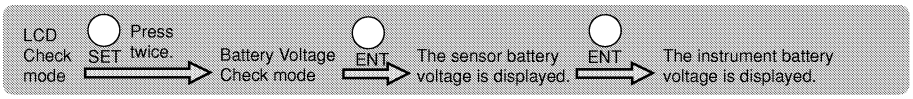
Standard solution ℃	160-22 Phthalic-acid chloride + quinhydrone	160-51 Neutral phosphate + quinhydrone
5	+274.2	+111.9
10	+270.9	+106.9
15	+266.8	+101.0
20	+262.5	+95.0
25	+257.6	+89.0
30	+253.5	+82.7
35	+248.6	+76.2
40	+243.6	+69.0

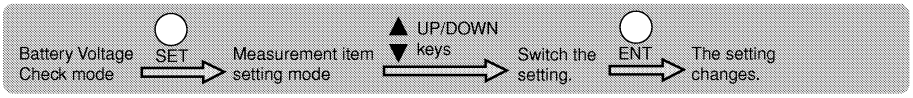
6. Using the various functions

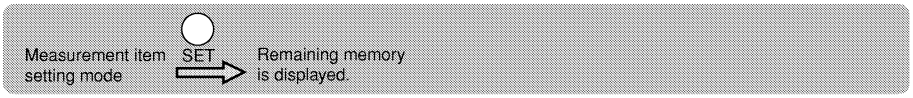


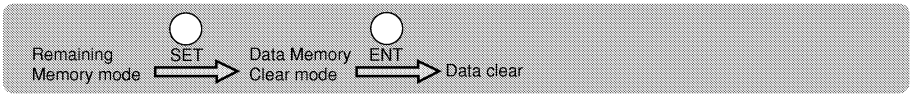


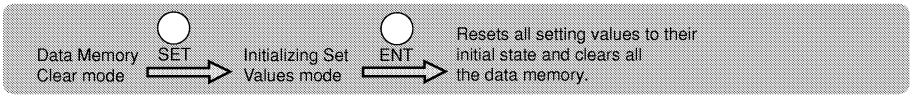


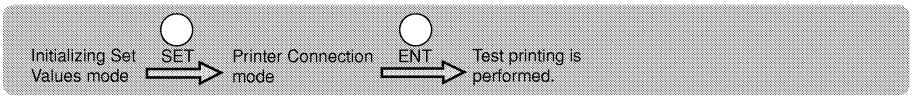






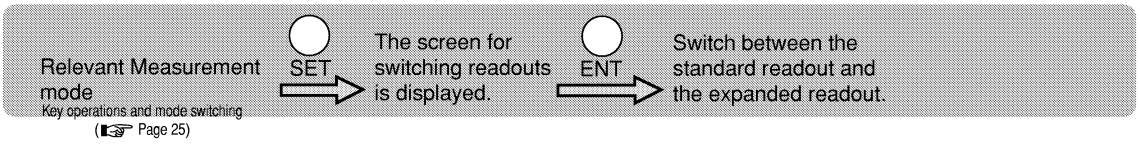






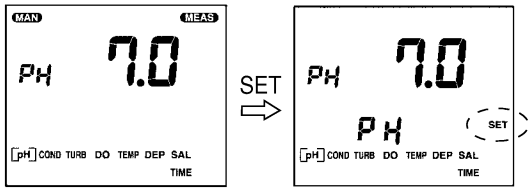
6.1 Switching to Expanded readout (High-accuracy display)

With the exception of oxidation-reduction potential (ORP), it is possible to switch between the Standard readout and the Expanded readout for the measurement value.



1. Press the **SET** key in the relevant Measurement mode.

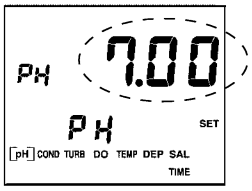
The screen for switching readouts is displayed.



Standard readout

2. Press the **ENT** key.

The screen can be switched between the standard readout and the expanded readout (High-accuracy display).



Expanded readout (High-accuracy display)

Note

- Switch readouts for each measurement items.
- Use the manual 2-point calibration (zero and span) when high accuracy is required for expanded readout (High-accuracy display).
- The expanded readout mode is automatically activated when the manual 2-point calibration mode is chosen.

3. Press the **MEAS** key to return to the Measurement mode.

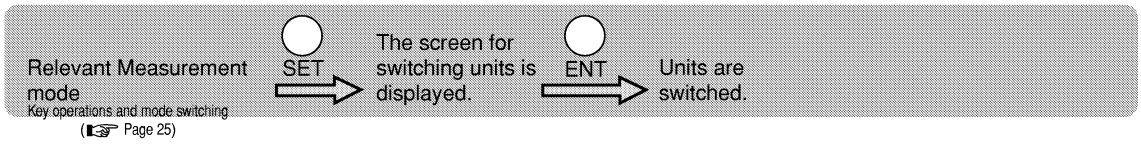
6.2 Switching measurement units

It is possible to switch between measurement units.

The units which can be switched are as follows:

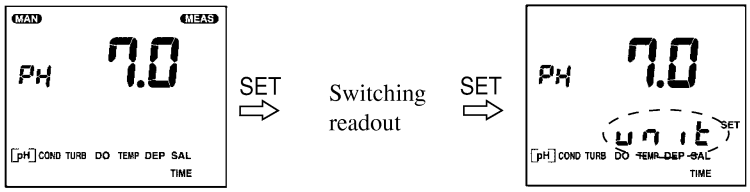
pH ..... pH or mV
Conductivity (COND) ..... S/m or mS/cm
Water depth (DEP) ..... m or ft
Turbidity (TURB) ..... NTU or mg/L
Dissolved Oxygen (DO) ..... mg/L or % (Oxygen saturation ratio)

In the case of pH, TURB, DO



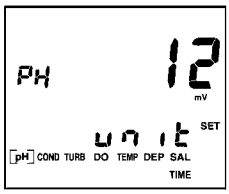
1. Press the **SET** key twice in the relevant Measurement mode.

Confirm that unit is displayed on the screen for switching units.



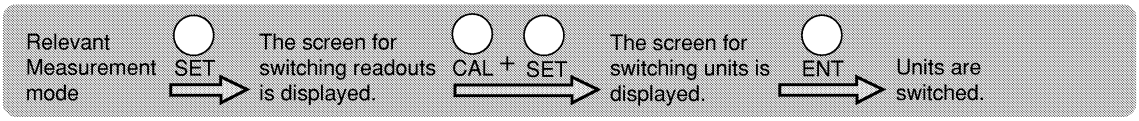
2. Press the **ENT** key.

Units are switched.



3. Press the **MEAS** key to return to the Measurement mode.

In the case of COND and DEP



1. Press the **SET** key in the Relevant Measurement mode.

The screen for switching readout is displayed.

2. Press the **SET** key while holding down the **CAL** key.

Confirm that **unit** is displayed on the screen for switching units.

3. Press the **ENT** key.

Units are switched.

4. Press the **MEAS** key to return to the Measurement mode.

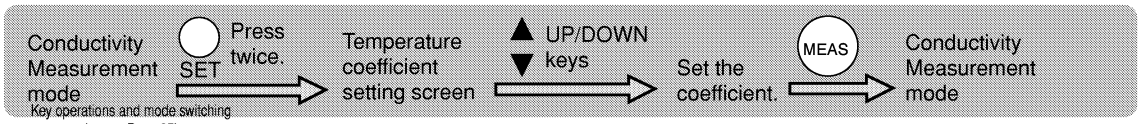
Measurement range

Measurement item		Measurement range		Measurement units
		Expanded	Standard	
pH		0.00 to 14.00	0.0 to 14.0	pH
		—	−1999 to 1999	mV in pH measurement
Conductivity (COND)	Range 1	0.90 to 9.99	0.9 to 9.9	S/m
		9.0 to 99.9	9 to 99	mS/cm
	Range 2	0.090 to 0.999	0.09 to 0.99	S/m
		0.90 to 9.99	0.9 to 9.9	mS/cm
	Range 3	0.0 to 99.9	0 to 99	mS/m
		0.000 to 0.999	0.00 to 0.99	mS/cm
Turbidity (TURB) *1		0.0 to 800.0	0 to 800	NTU (nephelometric turbidity units) or mg/L
Dissolved-oxygen (DO)		0.00 to 19.99	0.0 to 19.9	mg/L
		0.0 to 199.9	0 to 199	%
Temperature (TEMP)		0.00 to 55.00	0.0 to 55.0	°C
Water depth (DEP)		0.0 to 100.0	0 to 100	m
		0.0 to 330.0	0 to 330	ft
Salinity (SAL)		0.00 to 4.00	0.0 to 4.0	%
Total dissolved solids	Range 1	5.5 to 65.0	5 to 65	g/L
(TDS) *2	Range 2	0.55 to 6.50	0.5 to 6.5	g/L
	Range 3	0.000 to 0.650	0.00 to 0.65	g/L
Seawater specific gravity (σ_t)		0.0 to 50.0	0 to 50	–
Oxygen-reduction potential (ORP)		—	−1999 to 1999	mV

*1: Depending on the concentration range, the minimum turbidity is displayed as follows:
0 to 100 NTU ... 1 NTU for standard readout, 0.1 NTU for expanded readout.
100 to 800 NTU ... 10 NTU for standard readout, 1 NTU for expanded readout.
*2: The TDS range depends on the TDS factor settings. (Above ranges are given for a TDS coefficient of 0.65.)

6.3 Temperature conversion for conductivity (COND)

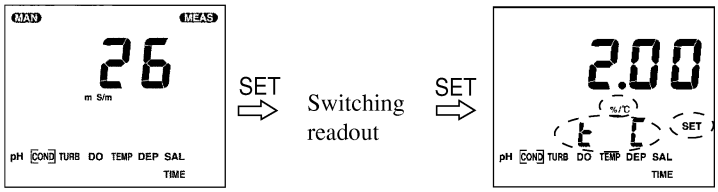
Sample conductivity (COND) varies with temperature, and this instrument uses a temperature conversion coefficient to automatically standardize the conductivity (COND) to the value at 25 °C. The initial setting value is 2 %/°C, which is the generally used value.



Key operations and mode switching (☞ Page 25)

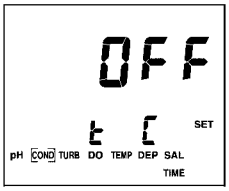
1. Press the **SET** key twice in the Conductivity (COND) Measurement mode.

The screen for setting temperature coefficients is displayed.



2. Use the **UP/DOWN** (▲ ▼) keys to set the coefficient.

The setting range is 0.00 to 3.00 %/°C.



With the ENT key, the temperature conversion is switched between ON and OFF.
The temperature conversion OFF mode is not a 25°C coefficient but a coefficient at the temperature of the sample.

3. Press the **MEAS** key.

The instrument returns to the Conductivity (COND) Measurement mode.

Note

- For temperature coefficients, refer to *Reference data*, page 98 to 99.

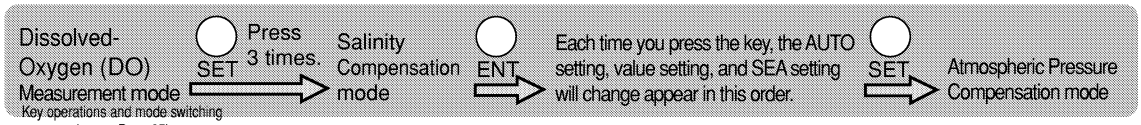
6.4 Dissolved-Oxygen (DO) environmental influence compensation

6.4.1 Salinity compensation

The indicated dissolved oxygen (DO) value can go over the actual value if salinity compensation isn't added because of the increase in salinity in the sample. To obtain a correct measured value for dissolved oxygen (DO) in the sample containing salinity, therefore, salinity compensation is needed. The following modes are available for calculation of salinity compensation.

AUTO..... Salinity compensation is performed automatically with salinity converted from a measured value for conductivity.

SEA ..... Compensation value appropriate for normal seawater is used.



Key operations and mode switching (☞ Page 25)

1. Press the **SET** key 3 times in the Dissolved-Oxygen (DO) Measurement mode.

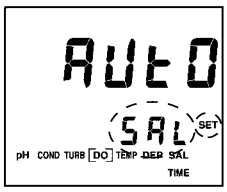
The salinity compensation mode currently set is displayed.

Important

- If you do not change the salinity compensation mode currently set, press the MEAS key to return to the Dissolved-Oxygen (DO) Measurement mode or press the SET key to select the Pressure Compensation mode.

2. Press the **ENT** key.

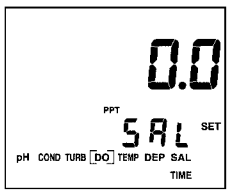
The following screens are displayed in sequence each time the ENT key is pressed: AUTO setting, value setting, SEA setting and AUTO setting.



3. From the screen on which the value is displayed, use the **UP/DOWN** (▲ ▼) keys to enter the setting value if the salinity is known.

For AUTO and SEA setting, this step need not be performed.

The setting range is 0.0 to 40.0 ppt (parts per thousand).

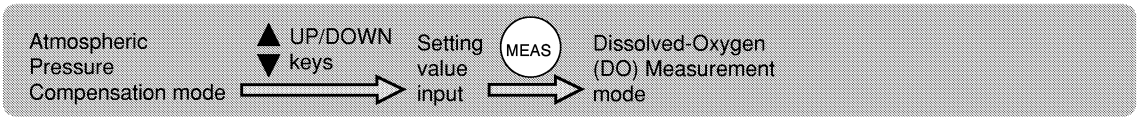


4. When the **SET** key is pressed, setting will be completed and the instrument will enter the Pressure Compensation mode.

5. Press the **MEAS** key to return to the Dissolved-Oxygen (DO) Measurement mode.

6.4.2 Atmospheric pressure compensation

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 instrument, it is possible to standardize the measured Dissolved-Oxygen (DO) value to a value at the standard atmospheric pressure (1013 hPa).



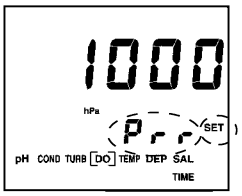
1. When the **SET** key is pressed on the salinity compensation screen, setting will be completed and the instrument will enter the Pressure Compensation mode.

Important

- If you do not change the Pressure Compensation mode currently set, press the MEAS key to enter the Dissolved-Oxygen (DO) Measurement mode.

2. Use the **UP/DOWN** (▲ ▼) keys to input a setting value.

The setting range is 100 to 1999 hPa.



3. When the **MEAS** key is pressed, setting will be completed and the instrument will enter the Dissolved-Oxygen (DO) Measurement mode.

Relation between height (m) and atmospheric pressure (hPa)

Height (m)	0	200	400	600	800	1000	1200	1400	1600	1800	2000	3000	3400
Pressure (hPa)	1013	990	966	943	921	899	877	856	835	815	795	701	666

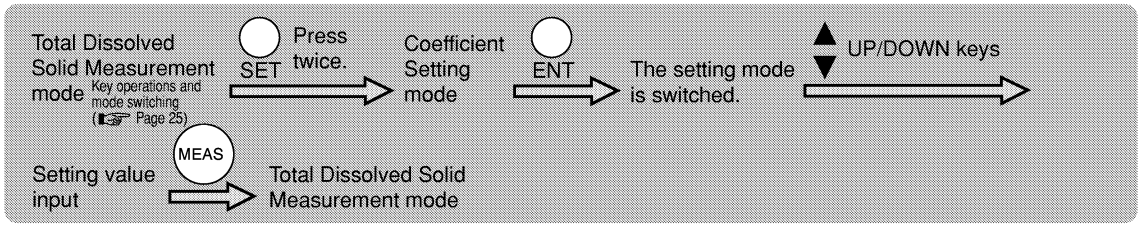
6.5 Setting a total dissolved solid (TDS) coefficient

The total dissolved solid amount (TDS) is a converted value obtained by multiplying the conductivity (COND) value by a known coefficient. Based on a conversion for KCl and $CaCO_3$ solutions, the coefficient initially set for the instrument 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

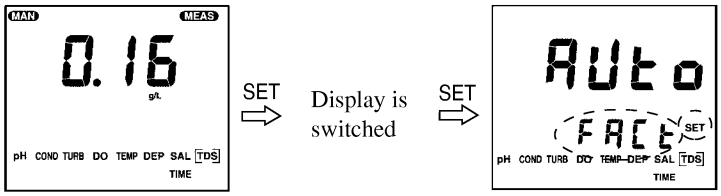
AUTO ..... Used to automatically calculate the total dissolved solid (TDS) amount with an initially set coefficient.

Setting value input ..... Used to determine the total dissolved solid (TDS) amount by setting any conversion coefficient irrespective of the conductivity (COND) value.



1. Press the **SET** key twice in the Total Dissolved Solid (TDS) Measurement mode.

The Coefficient Setting mode currently set is displayed.

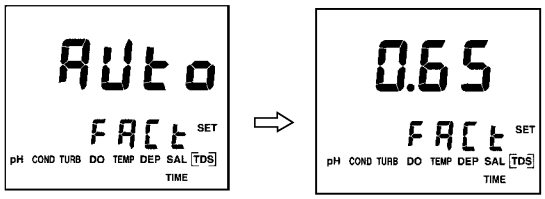


Important

- If you do not change the coefficient currently set, press the MEAS key to enter the Total Dissolved Solid (TDS) Measurement mode.

2. Press the **ENT** key.

The setting mode changes (AUTO/setting value input).



3. Use the **UP/DOWN** (▲ ▼) keys to input a setting value if required.

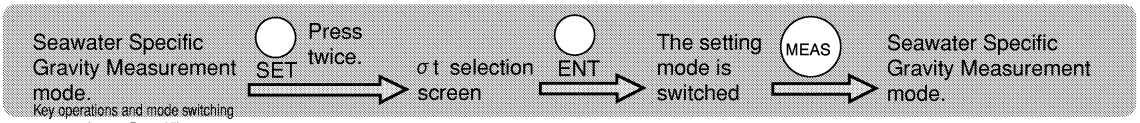
The setting range is 0.50 to 1.00.

4. When the **MEAS** key is pressed, setting will be completed and the instrument will enter the Total Dissolved Solid (TDS) Measurement mode.

6.6 Displaying seawater specific gravity (σ_0, σ_{15})

The specific gravity of seawater varies with temperature. By converting the measured value based on the value for a reference temperature, it is possible to compare sample measurement values at different temperatures.

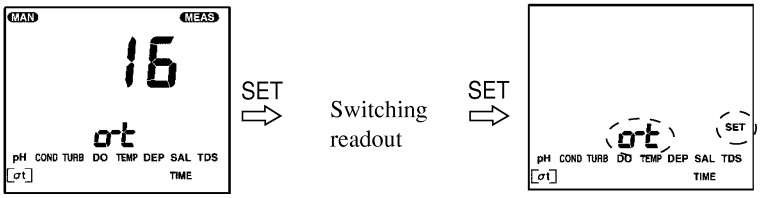
σ_t ..... Specific gravity of seawater at the measurement temperature.
σ_0 ..... Seawater specific gravity at 0 °C.
σ_{15} ..... Seawater specific gravity at 15 °C.



Key operations and mode switching
(☞ Page 25)

1. Press the **SET** key twice in the Seawater Specific Gravity (σ_t) Measurement mode.

Seawater specific gravity (σ_t) selection screen is displayed.

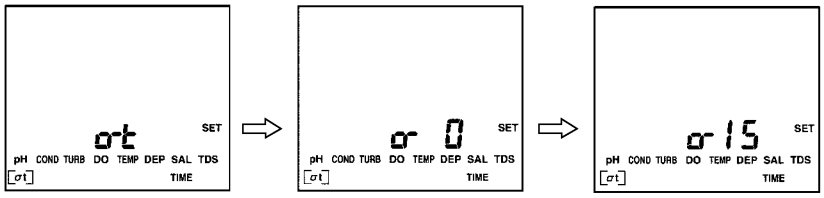


Important

- If you do not change the specific gravity currently set, press the MEAS key to enter the Seawater Specific gravity (σ_t) Measurement mode.

2. Press the **ENT** key.

The setting mode is switched.
($\sigma_0 \rightarrow \sigma_{15} \rightarrow \sigma_t \rightarrow \sigma_0 \ldots$)

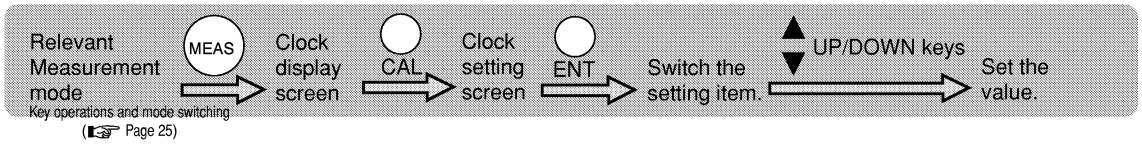


3. When the **MEAS** key is pressed, setting will be completed and the instrument will enter the Seawater Specific Gravity (σ_t) Measurement mode.

Note

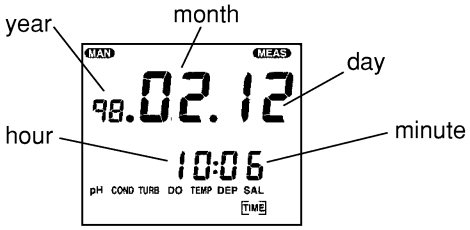
- See page 100 for more about seawater specific gravity.

6.7 Setting the clock



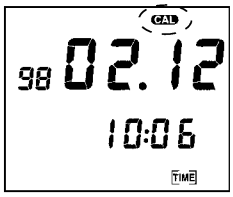
Key operations and mode switching (☞ Page 25)

1. Use the **MEAS** key in the measurement mode to switch to the clock display screen.



2. Press the **CAL** key.

CAL light up and clock setting screen is displayed.



3. Press the **ENT** key to switch the measuring item.

(year → month → day → hour → minute → year ...). The setting item will blink.

4. Use the **UP/DOWN** (▲ ▼) keys to set the value.

5. Press **SET** key to confirm the setting.

Note

- When the MEAS key is pressed, the instrument will return to the clock display.

Important

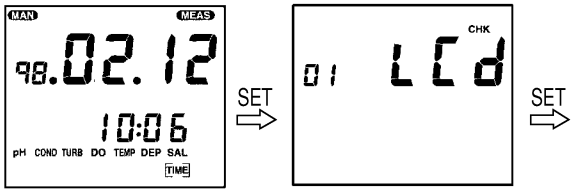
- When the MEAS key is pressed without pressing the SET key and the clock display is displayed again, settings are not changed.

6.8 Key lock setting

If you press the POWER key while pressing the UP (▲) key when the power is off, the instrument is then turned ON with the key locked and the key lock function works.
With the key locked, only the POWER and MEAS keys can be used and [LOCK] is displayed on the screen. To release this function, turn the instrument OFF first and then ON again.

6.9 Check mode

When the SET key is pressed in the measurement mode from the screen where "year, month, day and time" are displayed, the instrument performs self-diagnosis check.



Each time the SET key is pressed, the check mode item is switched sequentially.

Check mode items

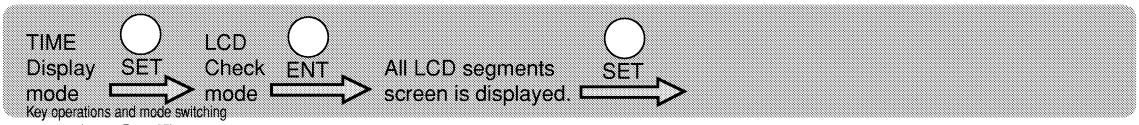
1 :	LCD check	Checks if all LCD segments will be displayed. (☞ Page 74)
2 :	Battery voltage check	Performs a simple battery voltage check for the instrument and sensors. (☞ Page 75)
3 :	Measurement item setting	Can set the measurement item to be stored. (☞ Page 76)
4 :	Remaining memory	Displays the number of data that can be stored now. (☞ Page 77)
5 :	Data memory clear	Clears the data memory. (☞ Page 78)
6 :	Initializing set values	Initializes all memory settings. (☞ Page 79)
7 :	Printer connection, test print	Performs a test print. (☞ Page 80)

Note

- In the check mode, it is possible to return to the Measurement mode by pressing the MEAS key.

6.9.1 LCD check

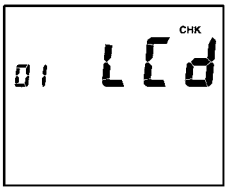
All LCD segments are displayed.



Key operations and mode switching
(☞ Page 25)

1. Press the **SET** key in the Clock Display mode.

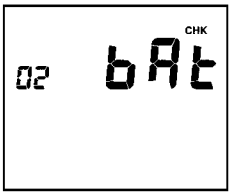
LCD check mode screen is displayed.



2. Press the **ENT** key.

3. Check to see if all LCD segments are displayed.

4. When the **SET** key is pressed, the instrument goes to the battery voltage check.

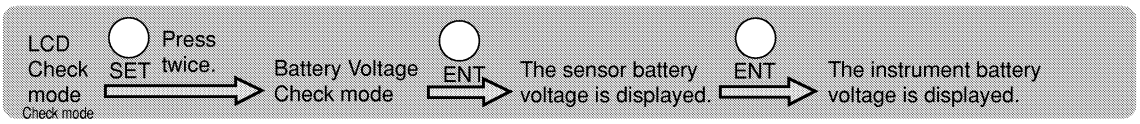


Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.9.2 Battery voltage check

The battery voltage in use is displayed.



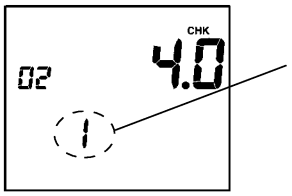
Check mode
(☞ Page 73)

1. Press the **SET** key twice in the LCD Check mode.

Battery Voltage Check mode screen is displayed.

2. Press the **ENT** key.

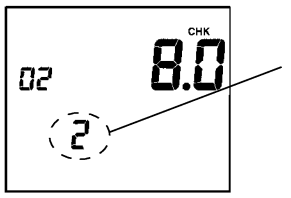
The sensor battery voltage is displayed.



1: Sensor battery voltage
Criteria
3.0 to 5.0 V : Normal
Less than 3.0 V: Replace the battery.
(Error No. 2 will blink.)

3. Press the **ENT** key.

The instrument battery voltage is displayed.



2: Instrument battery voltage
Criteria
5.5 to 11.0 V : Normal
Less than 5.5 V : Replace the battery.
(Error No.3 will blink.)

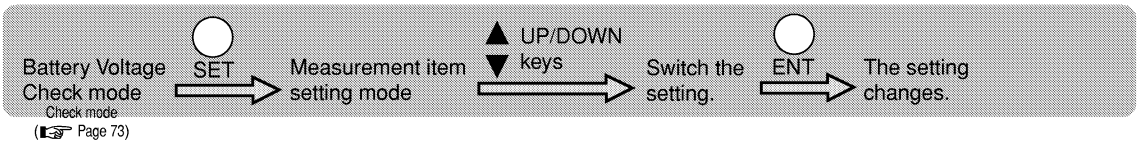
4. When the **SET** key is pressed, the instrument goes to the measurement item setting.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.9.3 Measurement item setting

Measuring items can be set.



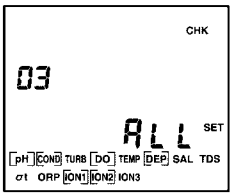
Check mode
(☞ Page 73)

1. Press the **SET** key in the Battery Voltage Check mode.

Display setting mode screen is displayed.

2. Use the **UP/DOWN** (▲ ▼) keys to switch the measurement item.

The selected item blinks.



3. Press the **ENT** key to switch between [set/ not set] for the blinking item.

An item for which "set" is selected is indicated with [ ].

Note

- If the temperature is "not set" data for each component is not temperature-compensated and is displayed as data at 25 °C.

4. When the **SET** key is pressed, the instrument goes to the remaining memory display.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.9.4 Remaining memory

The number of date that can be stored can be displayed.

Press the **SET** key in the Display Setting mode.

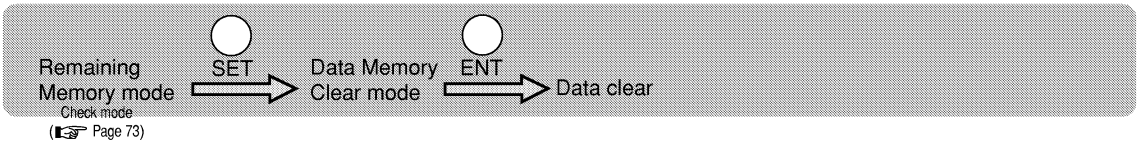
Remaining memory is displayed.

Note

- When the **SET** key is pressed, the instrument goes to the Data Memory Clear mode.
- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.9.5 Data memory clear

All the data memory is cleared.

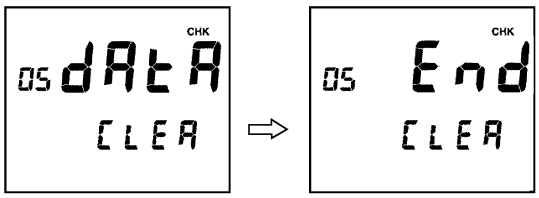


1. Press the **SET** key in the Remaining Memory mode.

Data memory clear mode screen is displayed.

2. Press the **ENT** key.

The data is cleared.



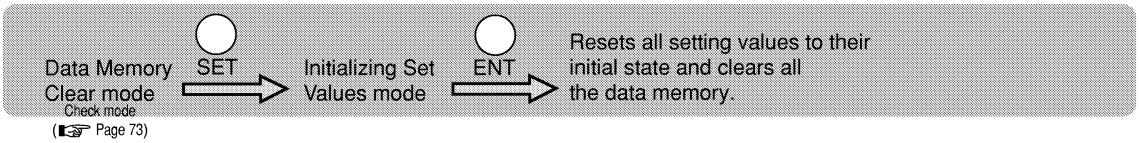
3. When the **SET** key is pressed, the instrument goes to the Memory Initialization mode.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

6.9.6 Initializing set values

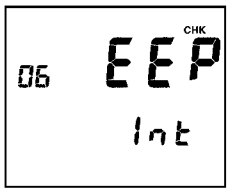
All setting values are reset to their initial state.



Check mode (☞ Page 73)

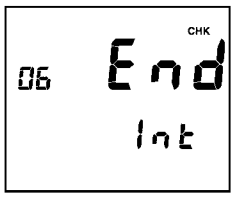
1. Press the **SET** key in the Data Memory Clear mode.

Initializing Set Values mode screen is displayed.



2. Press the **ENT** key.

All setting values are reset to their initial state.



3. When the **SET** key is pressed, the instrument goes to the Printer Connection mode.

Note

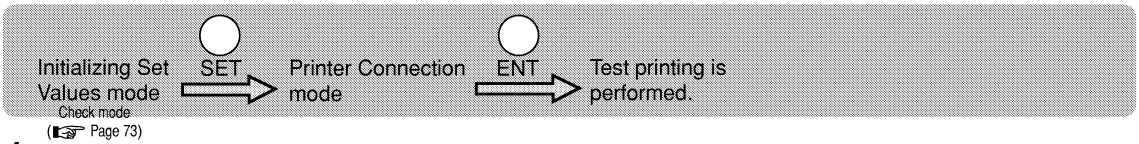
- When the MEAS key is pressed, the instrument return to the Clock Display mode.
- Data stored in the memory remains.

Initial setting

Item	Description	Initial value
Common	Display setting	Standard
	Data storage	Manual
pH	Unit	pH
COND	Unit	S/m
	Temperature coefficient	2.0 %/°C
DO	Salinity setting	AUTO
	Atmospheric pressure setting	1013 hPa
	Unit	mg/L
TURB	Unit	NTU
DEP	Unit	m
TDS	Coefficient	AUTO
σ_t	Unit	σ_t

6.9.7 Printer connection, test print

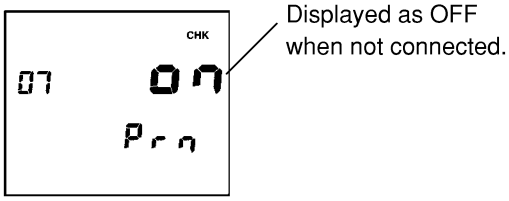
This mode only operates when the function expansion unit is connected. A test print is performed if a printer is connected.



Check mode (☞ Page 73)

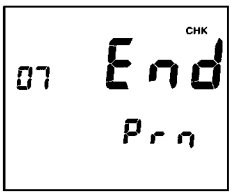
1. Press the **SET** key in the Initializing Set Values mode.

Printer Connection mode screen is displayed.



2. Press the **ENT** key to start printing.

Normally, "End" is displayed. If an error has occurred, "Err" is displayed.



3. When the **SET** key is pressed, the instrument will return to the first LCD check mode.

Note

- When the MEAS key is pressed, the instrument returns to the Clock Display mode.

7. Instrument specifications

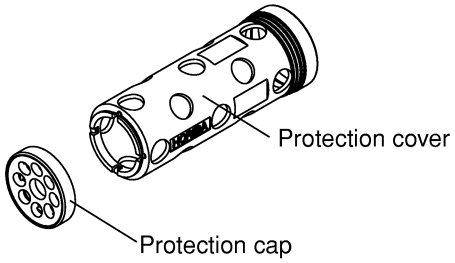
7.1 Daily maintenance

Sensor probe

● Storage method

After use, wash out with tap water and wipe off all contamination. Pour about 20 mL of pure water into the probe cap, install it on the sensor probe, and store in the carrying case.
In order to use the instrument regularly for a long time, store it after wiping off all contamination from the cable, sensor probe, and sensors.

Remove the protection cover once and completely wash out with tap water the left over sample on the screws. Reinstall the cover after having wiped off the drops of water. If there is any sample (especially sea water) left over on the screws, rust may form which may prevent the protection cover from being removed. (☞ Installation procedure, page 18.)
Depending on the level of contamination, remove the rubber protection cap from the tip of the protection cover and wash out with tap water. Reinstall it after wiping off the drops of water.

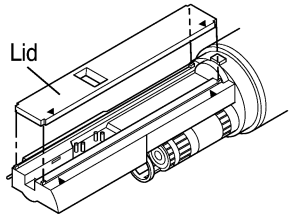


When storing with the pH/ORP and DO sensors attached to the probe, make sure to install the probe cap after having poured pure water into it.
Letting the pH/ORP and DO sensors get dry may cause deterioration of the instrument's performance. Should the sponge inside the probe cap be contaminated, replace it with a clean sponge (included).

TEMP/COND/TURB units

● To remove contamination

1. Remove the lid from the cell.
2. Clean the unit in tap water. If the unit is severely contaminated, use an absorbent cotton to remove contamination.
3. Attach the lid to the cell block before storage. (☞ page 29)



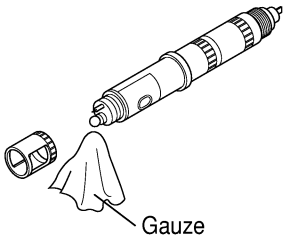
Important

- The cell has a window for turbidity measurement. Be careful to avoid damage to the window.
  In case of measurements, attach the lid to the cell in the correct direction.
- Don't remove the COND/TURB lid during calibration or measurement.
- Attach the lid to the cell with fitting four corners and facing ▲ marks each other.

pH/ORP sensors

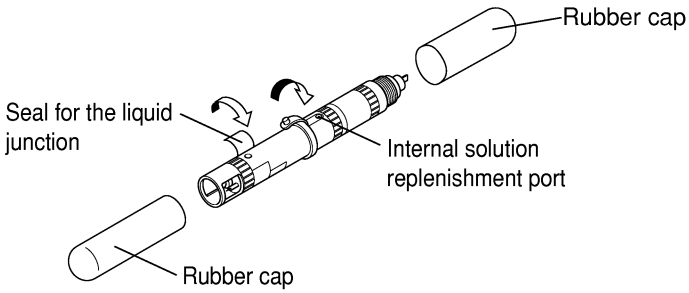
● To remove contamination

Use a piece of gauze dampened with detergent and wipe off contamination.



● Long-term storage

Remove the sensor from the sensor probe and check the internal solution replenishment port is closed. Then, attach a seal to the liquid junction and attach the rubber caps before storage.



● Monthly maintenance

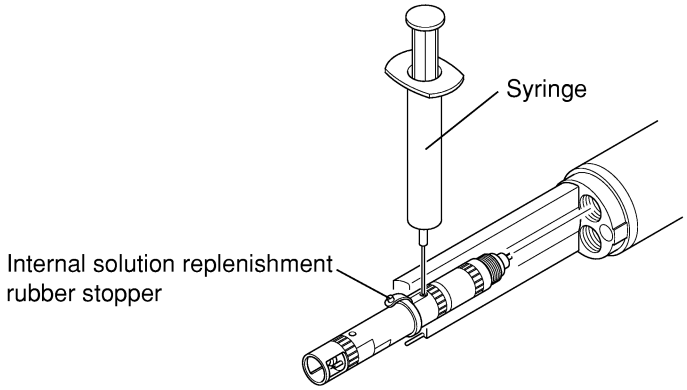
Replace the internal solution as described below:

1. Remove the sensor from the sensor probe using a sensor spanner.
2. Open the internal solution replenishment rubber stopper and remove the internal solution with a syringe.
3. Pour new internal solution (#330) to the level near rubber stopper. Be careful to avoid air bubbles from coming in the solition.
   Air bubbles in the internal solution will impair the sensors' pressure compensation function.

Important

- Shake the sensor to avoid bubbles in the internal solution from remaining at the bottom of the sensor.

4. Attach the sensor to the sensor probe.



DO sensor

● To remove contamination

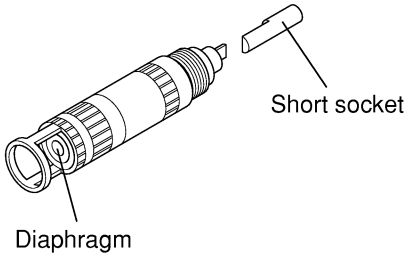
Wipe off contamination with gauze to avoid damage to the diaphragm.

● Long-term storage

Remove the sensor from the sensor probe using a sensor spanner. Set the supplied short socket and store the sensor in a cool(0 to 10℃), dark place.

Important

- Provide the DO sensor with a short socket or connect the sensor to the sensor probe for storage. Otherwise, the sensor may have a shorter life or stable instructions may not be obtained.
- The short socket is used when storing. Do not throw it away.



● Resetting the DO sensor when storing without having installed the short socket.

When leaving the DO sensor unattended for a brief period (1 or 2 days) without the short socket, the DO sensor can be reset by connecting it to the short socket or the probe. However, an amount of time corresponding to the period it was left unattended is necessary. If left unattended without being connected to the short socket or the probe for a long period (1 month), it cannot be reset.

● To replace the diaphragm.

Please read the instruction manual of the DO diaphragm replacement kit. (☞ page 89)

7.2 Troubleshooting

The instrument has a simple error message that informs users of operational errors and failure. Err No. is displayed at the bottom of the screen.

● Error message list

Err No.	Designation	Err No.	Designation
1	Sensor memory failure	6	Span calibration error
2	Sensor battery voltage drop	7	Calibration stability error
3	Instrument battery voltage drop	8	Printer error
4	Communications error	9	DATA IN error
5	Zero calibration error		

● Error and remedy

Important

- For err Nos. 5 to 7, the calibration err display disappears when a proper calibration is performed after the following action, or when the instrument is turned on again. For the other err Nos., the err display disappears after any of the following actions is taken.
- Error Nos. 2 and 3 are displayed even when using the AC adapter if the sensor probe battery voltage or instrument battery voltage drops is low on voltage.

Err NO.	Problem	Cause	Remedy
1	No data can be read from or written into the sensor probe memory.	Internal IC failure	Call your nearest store for sensor probe repair.
2	Sensor probe battery voltage drop	① Battery voltage drop ② Improper installation of the battery	① Replace the sensor probe battery. ② Set the batteries (LR03) in the correct direction.
3	Instrument battery voltage drop	① Battery voltage drop ② Improper installation of the battery	① Replace the instrument battery. ② Set the battery (6LR61) in the correct direction.
4	No communications possible between the instrument and the sensor probe	① Improper connection of the connector to the instrument ② Cable disconnection	① Connect the connector to the instrument properly and turn on the instrument again. ② Call your nearest store for cable repair.
5	No zero calibration possible	pH • The standard solution is contaminated. • Contamination on the pH glass membrane • Change in concentration of the internal solution for the reference electrode • Cracks in the pH glass electrode COND • The standard solution is contaminated. • The sensor is dirty. • The COND sensor is broken.	pH • Change the standard solution. • Clean the pH glass membrane. • Replace the internal solution for the reference electrode. • Replace the sensor. COND • Change the standard solution. • Clean the sensor. • Contact your nearest store.

Err NO.	Problem	Cause	Remedy
5	Zero calibration not possible	TURB	TURB
		• Air bubbles in the cell	• Swing the sensor probe while drawing a large arc.
		• Cell contamination	• Clean the cell.
		DO	DO
		• Damage to the diaphragm of the DO sensor	• Check the sensor and replace it if damaged.
		DEP	DEP
		• Contamination on the DEP sensor	• Clean the DEP sensor.
		• Damage to the DEP sensor	• Contact your nearest store.
6	Span calibration not possible	pH	pH
		• Contamination on the pH glass membrane	• Clean the pH glass membrane.
		• Change in concentration of the internal solution for the reference electrode	• Replace the internal solution for the reference electrode.
		• Cracks in the pH glass electrode	• Replace the sensor.
		• Damage to the connector pin	• Replace the sensor.
		COND	COND
		• The standard solution isn't correct.	• Calibrate with correct standard solution.
		• The standard solution value is set uncorrectly.	• Delete the calibration data for the conductivity, then calibrate the sensor again. (☞ Page 50)
		• The COND sensor is broken.	• Contact your nearest store.
		TURB	TURB
		• Air bubbles in the cell	• Swing the sensor probe while drawing a large arc.
		• Cell contamination	• Clean the cell.
		• The lid is attached uncorrectly.	• Confirm if the lid is attached correctly, then calibrate the sensor again. (☞ Page 29)
		DO	DO
		• Damage to DO sensor diaphragm	• Check the DO sensor and replace it if damaged.
		• DO sensor is unstable.	• Connect DO sensor to the sensor probe. Calibrate the sensor again 1 day later.
		• Damage to the connector pin	• Replace the sensor.
		DEP	DEP
		• Contamination on the DEP sensor	• Clean the DEP sensor.
		• Damage to the DEP sensor	• Contact your nearest store.
		TEMP	
		• Damage to the TEMP sensor	• Contact your nearest store.
7	The calibration value does not become stable within approximately three minutes.	① Sensor contamination	① Clean each sensor.
		② Dry sensor surface	② Pour the standard solution into the calibration beaker. Calibrate the sensor again 1 to 2 hours later.
		③ Severe temperature change	③ Calibrate the sensor in a place at a stable temperature or in a thermostatic oven.

Err NO.	Problem	Cause	Remedy
8	Printer unit failure		Turn OFF the instrument and use the remedy described below. Then turn ON the printer again.
		① Paper has jammed in the printer	① Remove the jammed sheet of paper
		② Improper printer unit connection	② Check to see if the printer is properl connected to the instrument.
		③ Printer failure	③ Replace the printer.
			* Contact your nearest store if the instrument does not recover after replacement of the printer.
9	Data cannot be stored because the memory is full.	No free space in the memory	Delete the data stored in the memory. (☞ Page 78)

● Other troubles

Remedies for various trouble with no Err No. displayed are described below.

Problem	Cause	Remedy
No data display with the power on	• No batteries	• Set new batteries.
	• Improper position of the positive and negative poles	• Set the batteries properly while paying attention to the positive and negative poles.
	• Battery voltage drop	• Replace the batteries with new ones.
	• Improper instrument battery contact	• Use radio pliers to narrow the positive terminal of the battery snap.
No setting change possible	• Automatic data storage is under way	• Press the CAL key to stop the automatic data storage.
No key operation possible	• The key lock function is working	• Turn OFF the instrument. Then turn ON the instrument again. (☞ Page 73)
	• Failure to calibrate the sensor or wrong calibration.	• Calibrate the sensor properly.
Blinking measured value	• Improper measurement sample	• Use a sample that is in the measurement range.
	• Sensor contamination	• Clean each sensor.
	• Poor calibration is possible. (The standard solution is contaminated.)	• Carry out correct calibration.
tYPE Err The Err is displayed and the operation cannot be performed.	• Improper connection of the cable connector to the instrument	• Connect the connector to the instrument properly and turn on the instrument again.
	• Cable disconnection	• Contact your nearest store.
	• Instrument inside failure	• Contact your nearest store.

● Troubleshooting for the TURB sensor

If an abnormal value such as -10, 800 or more is indicated, or indication does not become stable, follow as below instructions.

Remove the contamination of the sensor

Remove the cover of the turbidity (TURB) sensor, and clean the sensor with cotton swab.
Contamination or bubbles on the sensor may cause fluctuation of TURB values.

Remove bubbles around the sensor

When immersing sensor in the calibration cup, be sure lower it slowly. Quick immersion may cause bubbles on the sensor, which can have bad influence on calibration accuracy to give abnormal value indication.

Use of new standard solution

When calibration, clean the sensor before immersing it in the new standard solution. In case of zero calibration, when the standard solution is turbid or contaminated, calibrate again with the new standard one.

Points to be noted in making measurement

Immerse the sensor slowly in the sample. In case of abnormal measurement value observed, contamination or bubbles adhering may be suspected. So, shake greatly the sensor. Since immersion of the sensor in the sludge layer at the bottom of the sample can prohibit accurate measurement, shake greatly enough to remove the sludge.

● Maintenance of DO sensor

Durable life of DO sensor is generally one year, however, it may vary depending on the using condition. In case of the failure of calibration or breakage of the diaphragm, take either of the following steps according to the using period.

Within one year after purchasing :

Obtain diaphragm replacement kit (optional) to replace the used diaphragm and replenish the internal solution for restoration.

When exceeding one year after purchasing :

Replace by the new DO sensor.

● Materials

#5460 DO Sensor Diaphragm Replacement Kit Operation Manual

This operation manual explains how to replace the DO Sensor (#5460) Diaphragm.

1 This kit comes with the following.

● Diaphragm sheets	10 sheets
● Diaphragm retaining ring	5 pcs.
● O−ring (S9)	5 pcs.
● Internal solution #305 (50 mL)	1 bottle
● Diaphragm retaining plate	1 pc.
● Replacement stand	1 pc.
● Syringe and needle	1 set
● Operation manual	1 sheet

2 Diaphragm Replacement

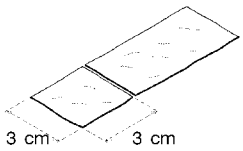
Chemical solution

Caution

The internal solution contains pottasium hydroxide (KOH) solution of high concentration.
If the solution comes in contact with hands or skin, wash immediately with water. If the solution comes in contact with the eyes, flush with ample amounts of water, then seek medical assistance.

1 Cut a diaphragm sheet to about 3 x 3 cm in size.

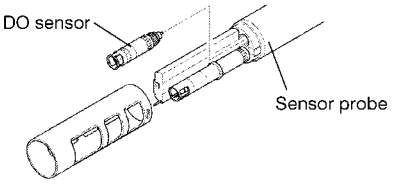
Note▶▶▶ Don't get any fingerprints or dust on the center part of the square.



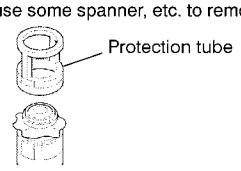
2 Detach the DO sensor from the sensor probe.

Ref▶▶▶ Refer to the U−21.22.23 Operation Manual, section 2.3.2 "Sensor Installation".

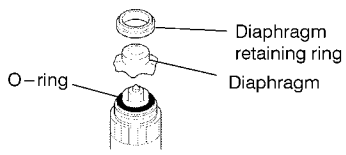
Note▶▶▶ If the short socket is not attached, instability may be seen when the sensor is used again.
Wear vinyl work gloves when handling the sensor. The KOH inside it is a strong alkaline and can irritate skin.



3 Detach the protection tube that holds the diaphragm in place. If it is difficult to remove, use some spanner, etc. to remove it.

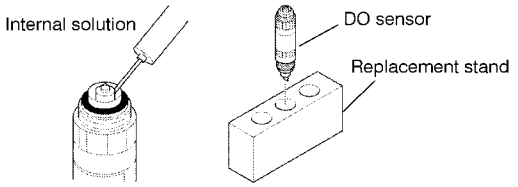


4 Detach the diaphragm retaining ring and diaphragm. Detach the diaphragm retaining ring and diaphragm if damaged or no longer functional.

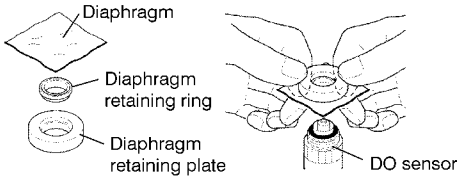


5 Set the sensor in the replacement stand ,and fill the internal solution with the attached syringe until the sensor tip is soaked with the solution.

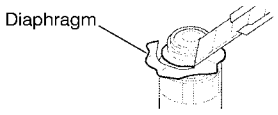
Note▶▶▶ Use the internal solution included in the kit for best performance.
In case some white crystals are seen, wipe them out completely with a Kimwipe®, etc.
In case the internal solution includes white crystals, remove the internal solution and fill up again. If the white crystals cannot be seen, just fill up.



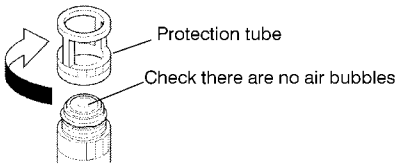
6 Fit the diaphragm retaining ring into the diaphragm retaining plate. Then, lay the diaphragm over the sensor and carefully cover with the ring and plate so that the diaphragm does not wrinkle. Finally, remove the retaining plate.



7 Cut the draped edge of the diaphragm to the shape of the sensor.

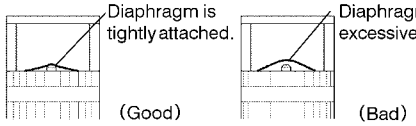


8 Check there are no air bubbles inside the sensor and tighten the protection tube securely.



9 Check the diaphragm bulges upward as shown on the below left and that it is not wrinkled.

Note▶▶▶ Check that span calibration can be made correctly. Sensitivity lowers and response speed slows if the diaphragm does not bulge sufficiently.



For any question regarding this product, please contact your local agency, or inquire from the Customer Registration website (www.horiba.co.jp/register).

HORIBA, Ltd.

7.3 Specifications

NOTE ○: Applicable
—: Unapplicable

		U-22XD
Instrument	Water-proof construction	IP67
	Mass	Approximately 500 g (including the grip holder)
Sensor *1	Use in 2-inch well	○
	Measurement temperature	0 to 55 °C
	Storage temperature	-5 to 60 °C
	Measurement depth	to 100 m
	Maximum sensor outside diameter	47 mm
	Sensor length	380 mm
	Continuous use available *2	30 days
	Automatic data gathering at set time	○
	Mass (Cable10 m)	Approximately 1.9 kg
pH ● Two-point calibration ● Automatic temperature compensation	Measuring principle	Glass electrode method
	Range	pH0 to 14
	Resolution	0.01 pH
	Repeatability	±0.05 pH
	Accuracy	±0.1 pH
Dissolved-Oxygen ● Salinity conversion (0 to 40 ppt/Auto) ● Automatic temperature compensation	Measuring principle	Diaphragm galvanic battery method
	Range	0 to 19.99 mg/L
	Resolution	0.01 mg/L
	Repeatability	±0.1 mg/L
	Accuracy	±0.2 mg/L
Conductivity ● Auto range ● Automatic temperature conversion (25 °C)	Measuring principle	4 AC electrode method
	Range	0 to 9.99 S/m
	Resolution	0.1 % of full scale
	Repeatability	±1 %
	Accuracy	±3 %
Salinity	Measuring principle	Conductivity conversion
	Range	0 to 4 %
	Resolution	0.01 %
	Repeatability	±0.1 %
	Accuracy	±0.3 %
Total Dissolved Solid(TDS) ● Conversion factor setting	Measuring principle	Conductivity conversion
	Range	0 to 99.9 g/L
	Resolution	0.1 % of full scale
	Repeatability	±2 g/L
	Accuracy	±5 g/L
Seawater specific gravity ● Display σ_t, σ_0, σ_{15}	Measuring principle	Conductivity conversion
	Range	0 to 50 σ_t
	Resolution	0.1 σ_t
	Repeatability	±2 σ_t
	Accuracy	±5 σ_t
Temperature	Measuring principle	Thermistor method
	Range	0 to 55 °C
	Resolution	0.01 °C
	Repeatability	±0.3 °C
	Accuracy	±1.0 °C

		U-22XD
Turbidity (TURB)	Measuring principle	Penetration and scattering method
● Unit selection	Range (NTU or mg/L)	0 to 800 NTU
	Resolution	0.1 NTU
	Repetability	±3 %
	Accuracy	±5 %
Water depth	Measuring principle	Pressure method
	Range	0 to 100 m
	Resolution	0.1 m
	Repetability	±3 %
	Accuracy	±5 %
Oxidation-reduction potential (ORP)	Measuring principle	Platinum electrode method
	Range	± 1999 mV
	Resolution	1 mV
	Repetability	± 5 mV
	Accuracy	±15 mV
Simultaneous measurement items		10

Note: The accuracy rating value is obtained from measurements at intermediate point of the standard solution after two-point calibration (at room temperature and pressure). The repeatability and accuracy rating percentages are based on the full scale (except for salinity).

*1: Organic solvents, strong acids, and strong alkaline solvents cannot be measured.

*2: Based on the data measured automatically at 15 minutes intervals. The battery life taken into account. Periodical maintenance and calibration is necessary when a lot of shellfishes and seaweeds exist at the measurement point.

7.4 Spare parts

Sensors

Sensor	Model	Spare part number
pH sensor	6230	9037-0056-00
pH/ORP sensor	6280	9037-0057-00
DO sensor	5460	9037-0058-00

Standard and internal solutions

Solution	Model	Spare part number	Remark
pH 4 standard solution (500 mL)	100-4	9003-0016-00	Standard solution for AUTO calibration, which is in addition used for manual pH span calibration.
pH 7 standard solution (500 mL)	100-7	9003-0017-00	Standard solution for pH zero calibration
pH 9 standard solution (500 mL)	100-9	9003-0018-00	Standard solution for manual pH span calibration
Powder for ORP standard solution (250 mL × 10)	160-51	9003-0031-00	Powdered standard solution to be used for checking ORP behavior
Powder for ORP standard solution (250 mL × 10)	160-22	9003-0030-00	
pH reference internal solution (250 mL)	330	9037-0052-00	Replenishment internal solution for pH reference electrode

Others

	Model	Spare part number	Remark
Calibration beaker XD	–	9037-0086-00	This is similar to the standard accessory, and used for sensor calibration.
Connector plug for the prove	–	9037-0071-00	When using the probe separately from the instrument, this is used to maintain waterproof of the connector.
Sensor spanner	–	9037-0088-00	This is used to connect the sensor to the probe. Similar to the standard accessory.
DO diaphragm replacement kit	–	9037-0074-00	In case of breakage of the DO sensor diaphragm, it is used in the replacement of the diaphragm to restore the sensor.
Battery cover packing	–	9096-0013-00	Replacement packing to be used for battery box of the main unit.
System unit cover O-ring	–	9096-0014-00	Replacement packing to be used for EXT cover of the main unit.
Sensor O-ring	–	9037-0076-00	Replacement O-ring to be used for connector of pH/ORP sensor and Do sensor.
Probe cap XD	–	9037-0087-00	This cap is to be used when storing the sensor probe.
Battery caver O-ring	–	9037-0084-00	This replacement O-ring is used for the sensor probe's battery cover.
Silicon grease	–	9037-0085-00	This silicon grease is applied on the sensors' O-rings. Similar to the standard accessory.
Protection cover packing	–	9037-0091-00	This is packing for when taking off the probe cap and seal after installing the protection cover. (board packing and O-ring set)
Sponge	–	9037-0089-00	This replacement sponge is used for the probe cap XD.
Protection cap	–	9037-0090-00	This cap is to be attached to the protection cover.

* The spare parts above are prepared by dealers.
Order the part by designating the parts name, model and spare parts number.

7.5 Option

Parts name	Model	Remark
Expansion adaptor	U-2001	This is applicable to AC adapter connection, RS-232C communication, GPS connection, printer output, and data-collecting software.
System unit *	U-2002-100V U-2002-110V U-2002-220V	This is applicable to AC adapter connection, RS-232C communication, GPS connection, printer output, and data-collecting software. GPS and printer are included in a complete set.
AC adaptor (for 100 V)	AC-10	AC adapter intended to drive the U-20 series by AC power supply. This should be used together with U-2001 and U-2002.
Carrying case	W-2010	Compact carrying case for cable below 10 m in length . Not large enough to hold flow cell or guard.
Carrying case	W-2030	Bigger-sized carrying case for cable exceeding 30 m in length. Large enough to hold flow cell.
Flow cell	W-2100	To be used for measurement at a pumping up sample.
Probe guard	W-2200	To be used for measurement at a location where there is a flow or a location with a thick sludge layer residing at the bottom.
PC connection cable	—	Nine-pin connection cable to PC.

* Specify the power source and voltage of the printer when ordering.

8. Reference Data

● pH measurement

1. Principle of pH measurement

U-20XD 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 comparison electrode. For more information, refer to JIS Z 8802 pH measurement method.

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 when the pH is measured must be recorded along with that pH value, even if a meter that has automatic temperature compensation is used. If the solution temperature is not recorded, the results of the pH measurement may be meaningless.

3. Types of standard solutions

When measuring pH, the pH meter must be calibrated using a standard solution. There are five kinds of standard solutions specified in "JIS 28802 pH measurement". For normal measurement, two of standard solutions with a 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 ..... 0.05 mol/L potassium hydrogen phthalate aqueous solution
(Phthalate)
pH 7 standard solution ..... 0.025 mol/L potassium dihydrogenphosphate, 0.025 mol/L sodium phospate aqueous solution
(Neutral phosphate)
pH 9 standard solution ..... 0.01 mol/L tetra-sodium boric acid aqueous solution
(Borate)

pH values of pH standard solutions at various temperatures settings.

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

4. Supplements for pH measurement

Pressure compensation diaphragm

U-20XD series can measure pH with high accuracy through the pressure compensation diaphragm without being affected by hydraulic pressure. Attention should be paid to the following points so that the diaphragm may function fully.
Before measurement, use a syringe and fill the reference electrode up to the replenish port with the internal solution. When injecting the polarity reference internal solution, be careful that air bubbles do not get into the solution.

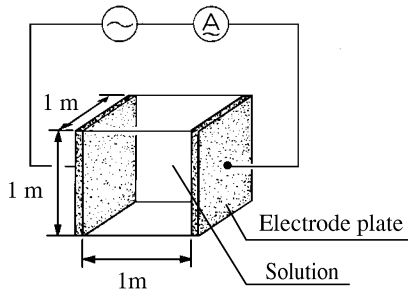
● COND measurement

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.

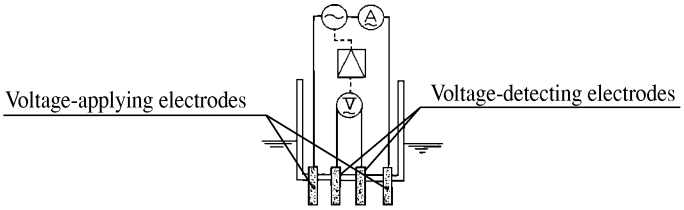
A cube with 1 m on each side, as shown in Fig. 1, is used to demonstrate an electrolytic solution. Two electrode plates are placed on opposite sides, and the cube is filled with a solution. If the resistance between these two electrode plates is represented by r (Ω), the conductivity of the solution L ($S.m^{-1}$) is represented as L=1/r. S stands for Siemens, a unit of measurement of conductance.



(Fig. 1 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-20XD series has adopted the 4-electrode method to overcome these disadvantages of the the 2-electrode method. As shown in Fig. 2, the U-20XD 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. 2 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 conductivity, 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.

2. SI units

New measurement units, called SI units, have been in use from 1996. Accordingly, the U-20XD 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 units
Measurement	0.1 mS/cm	→	0.01 S/m
value	1 mS/cm	→	0.1 S/m
	100 mS/cm	→	10 S/m

3. Temperature coeffcient

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 tempreture 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-20XD 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-20XD 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
(value displayed on U-20XD series)
t : Temperature of solution at time of measurement (˚C)
L_t : Conductivity of solution at t (˚C)
K : Temperature coeffcient (%/˚C)

Conductivity and temperature coefficient for various types of solutions

Conductivity and related temperature coefficients of representative substances (at 25 °C) are shown in the table below.

Substance	Concentration wt%	Conductivity S/m	Temperature coeffcient %/°C	Temperature °C
NaOH	5	19.69	2.01	15
	10	31.24	2.17	
	15	34.63	2.49	
	20	32.70	2.99	
KOH	25.2	54.03	2.09	15
	29.4	54.34	2.21	
	33.6	52.21	2.36	
	42	42.12	2.83	
NH_3	0.1	0.0251	2.46	15
	1.6	0.0867	2.38	
	4.01	0.1095	2.50	
	8.03	0.1038	2.62	
HCl	5	39.48	1.58	18
	10	63.2	1.56	
	20	76.15	1.54	
	30	66.20	1.54	
H_2SO_4	5	20.85	1.21	18
	10	39.15	1.28	
	20	65.27	1.45	
	40	68.00	1.78	
	50	54.05	1.93	
	60	37.26	2.13	
	100.14	1.87	0.30	
HNO_3	6.2	31.23	1.47	18
	12.4	54.18	1.42	
	31	78.19	1.39	
	49.6	63.41	1.57	
H_3PO_4	10	5.68	1.04	15
	20	11.29	1.14	
	40	20.70	1.50	
	45	20.87	1.61	
	50	20.73	1.74	
NaCl	5	6.72	2.17	18
	10	12.11	2.14	
	15	16.42	2.12	
	20	19.57	2.16	
	25	21.5	2.27	

Substance	Concentration wt%	Conductivity S/m	Temperature coeffcient %/°C	Temperature °C
Na_2SO_4	5	4.09	2.36	18
	10	6.87	2.49	
	15	8.86	2.56	
Na_2CO_3	5	4.56	2.52	18
	10	7.05	2.71	
	15	8.36	2.94	
KCl	5	6.90	2.01	18
	10	13.59	1.88	
	15	20.20	1.79	
	20	26.77	1.68	
	21	28.10	1.66	
KBr	5	4.65	2.06	15
	10	9.28	1.94	
	20	19.07	1.77	
KCN	3.25	5.07	2.07	15
	6.5	10.26	1.93	
NH_4Cl	5	9.18	1.98	18
	10	17.76	1.86	
	15	25.86	1.71	
	20	33.65	1.61	
	25	40.25	1.54	
NH_4NO_3	5	5.90	2.03	15
	10	11.17	1.94	
	30	28.41	1.68	
	50	36.22	1.56	
$CuSO_4$	2.5	10.90	2.13	18
	5	18.90	2.16	
	10	32.00	2.18	
	15	42.10	2.31	
CH_3COOH	10	15.26	1.69	18
	15	16.19	1.74	
	20	16.05	1.79	
	30	14.01	1.86	
	40	10.81	1.96	
	60	4.56	2.06	

● SAL conversion

The U-20XD series is designed to measure salinity as well as the other parameters.
Note that the "salinity" referred to 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 is known. In other words, the salinity measurement of the U-20XD 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, for example, hydrochloric acid (HCl).

● 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) × K × 10
	TDS(g/L) = L (mS/m) × K ÷ 100
Conductivity in the old units (mS/cm)	TDS(g/L) = L (mS/cm) × K
	K = TDS coefficient

Initial settings use the values listed in the table (☞ Page 70) 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.

● σ_t 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.

$$\sigma = (\rho - 1) \times 1000$$

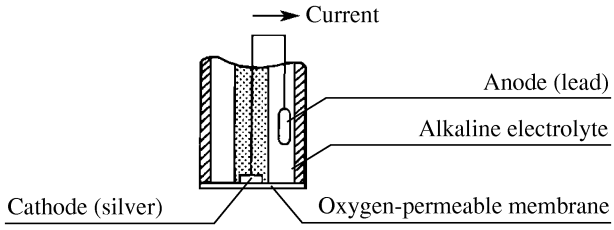
The density of seawater ρ is expressed by temperature, hydraulic pressure, and salinity functions. The density of seawater σ under the atmospheric pressure is expressed as σ_t. The density of seawater under the atmospheric pressure is determined by temperature and salinity.
The U-20XD series models make 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-20XD series models, newly added as measurement components.

● DO measurement

1. Principle of measurement

The "DO" referred to here means the concentration of oxygen dissolved in water. DO is essential to self-purification of river and sea and to water creatures such as fish. DO measurement is also essential to drainage and water quality control.

Fig. 3 shows the principle of measurement using a DO sensor.



(Fig. 3 Principle of DO sensor)

A noble metal (silver) is fitted closely to an oxygen-permeable membrane to make the cathode; a base metal (lead) is used as the anode. Both are immersed in an alkaline electrolyte with the anode-to-cathode external circuit closed. Oxygen diffusing through the oxygen-permeable membrane causes a reduction reaction at the cathode; this allows flow of current in the external circuit:

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$$

At the anode, oxidation reaction occur as follows:

$$2Pb \rightarrow 2Pb2^+ + 4e^-$$

The current is proportional to the quantity of oxygen diffusing through the oxygen-permeable membrane. Accordingly, measurement of the current makes the DO in a sample known.

The DO measuring method based on this principle is called the membrane-electrode method. This method allows convenient measurement of DO, especially when compared with chemical-analysis method, which needs complicated pre-treatment to eliminate the effects of oxidizing or reducing substances.

2. DO correction for salinity

When a solution and air are in contact and in complete equilibrium (saturated), DO: C [mg/L] in the solution, and the oxygen partial-pressure: Ps [MPa] in air are in the following relation:

$$C = Ps/H$$

H [MPa/ (mg/L)] is referred to as Henry's constant, which depends on the composition of the solution. In general, C becomes smaller as the salinity in the solution increases, since H becomes larger.

A DO sensor is intended to detect Ps in the above expression. Therefore, the DO measurement would be in error if the DO sensor were immersed either in air-saturated pure water or in solution with salt. To settle this problem, it is necessary to correct the DO reading based on the salinity of the sample using salinity correction.

Conventional DO meters make this salinity correction by inputting a known salinity value. This poses no problems if the salinity of the sample is known. In general, however, the salinity of the sample is usually not known, and the method is not practical even if the DO meters are equipped with the salinity correction function.

The U-20XD series is capable of measuring the salinity of a sample and automatically correcting the using this function.

3. Features of the U-20XD series DO sensors

In conventional DO measurements, it was necessary to keep the velocity of the flow constant because the velocity of flow led to fluctuation in indicated values. In our U-20XD series models, improvements in sensors have made it possible to make measurements with stable indications and with little influence of the velocity of flow.

● Turbidity (TURB) measurement

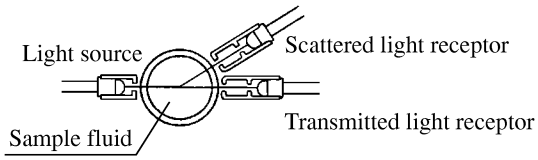
1. Principle of measurement

From among several types of turbidity-measuring methods available, the U-20XD series uses the light-transmission-scattering method, shown in Fig. 4.

Irradiation of a beam of light onto a sample brings about separation of the beam into (1) the light transmitted through the solution and (2) the light scattered by turbidity components in the sample. In the light-transmission-scattering method, the intensity of both transmitted light and the scattered light are measured using separate receptors, and the turbidity is obtained based on the ratio of the two.

With the U-20XD series, the light source is a pulse-lighting infrared-emission diode. The scattered light is measured at a point 60° offset from the light source. This light-absorption-scattering method has several advantages, including the fact that (1) the actual color of the sample fluid has little effect on the measurement of turbidity, (2) fluctuations in light quantity from the light source are easily compensated for, and (3) it allow the U-20XD series to be operated with relatively low-power consumption.

The turbidity value differs with the structure of the cell so changes with the instrument.



(Fig. 4 Principle of the light-transmission-scattering method)

2. Standard solution

U-20XD 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.

● DEP measurement

1. Depth (DEP) measurement

For the U-22XD model, 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.

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:

Immerse the depth sensor of the sensor probe into the sample.

Keep the sensor immersed in the sample for approximately 30 minutes until the temperatures of the sensor and the sample are the same.

Then make the zero calibration of the sensor manually. (☞ Page 57)

● Measuring mV (oxidation-reduction potential (ORP))

ORP principles

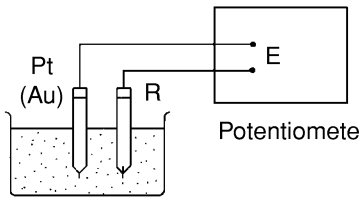
ORP (or "redox potential") is an abbreviation for oxidation-reduction potential. ORP is the energy level (potential) determined according to the state of equilibrium between the oxidants (M^{Z+}) and reductants $M^{(Z-N)+}$) that coexist within a solution.

$M^{z+} + ne^- \Leftrightarrow M^{(z-n)+}$.....①

If only ① exists within a solution, a metal electrode (platinum, gold, etc.) and a reference electrode are inserted into the solution, forming the ORP measuring system shown in Fig. 5. Measuring the potential (ORP) that exists between the two electrodes enables the potential to generally be expressed by the following equation.

$$E = E_0 - \frac{RT}{nF} \ln \frac{a_{M^{(z-n)+}}}{a_{M^{z+}}} \quad \text{..... ②}$$

E: Electric potential E_0 : Constant R : Gas constant T : Absolute temperature
n : Electron count F : Faraday constant a : Activity



(Fig. 5 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.

$Fe^{3+} + e^- \Leftrightarrow Fe^{2+}$.....①

$$E = E_0 - \frac{RT}{F} \ln \frac{a_{Fe^{2+}}}{a_{Fe^{3+}}} \quad \text{.....②}$$

When only one type of state of equilibrium 1 exists in the solution, the ORP of the solution can be determined uniquely by equation 2. What is important here is that ORP is determined by the ratio of activity between the oxidant (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 (potentiametric titration) and in the disposal and treatment of solutions.

Recently, there have appeared various claims regarding this matter, such as that a high degree of ORP is effective in sterilization or that drinking water that has a low ORP reduces the chance of illness by reacting with the activated oxygen in the cells of the body. ORP is used as an index for alkaline drinking water.

Standard electrode (reference electrode) types and ORP

The ORP of a solution that is obtained through measurement is a value that corresponds to the 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 uses Ag/AgCl with 3.33 mol/L KCl as the reference solution for reference electrodes. According to general technical literature, standard 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.

$E_{N.H.E.} = E + 206 - 0.7\,(t - 25)\text{mV} \qquad t = 0 - 60\,℃$

$E_{N.H.E.}$: 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 $Li^{+}+e^{-} \rightarrow Li$

E0=-3.024 V VS N.H.E.

However, in some literature, the "+" and "-" signs are reversed.

B $Li \rightarrow Li^{+}+e^{-}$

E0=+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.

表 3

For any question regarding this product,
please contact your local agency,
or inquire from the Customer Registration
website (www.horiba.co.jp/register).

HORIBA, Ltd.

First edition: November 2001
CODE : I1000908000

LaMotte

2020 TURBIDIMETER

Instruction MANUAL

TABLE OF CONTENTS

GENERAL INFORMATION

PACKAGING & DELIVERY

Experienced packaging personnel at LaMotte Company assure adequate protection against normal hazards encountered in transportation of shipments. After the product leaves the manufacturer, 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.

Should it be necessary to return the instrument for repair or servicing, pack the instrument carefully in a suitable container with adequate packing material. A return authorization number must be obtained from LaMotte Company by calling 1-800-344-3100 or faxing 1-410-778-6394. Attach a letter with the authorization number to the shipping carton which describes the reason for the return. This information will enable the service department to make the required repairs more efficiently.

GENERAL PRECAUTIONS

Read the instruction manual before attempting to set up or operate this instrument. Failure to do so could result in personal injury or damage to the instrument.

The 2020 Turbidimeter should not be stored or used in a wet or corrosive environment. Care should be taken to prevent water from wet turbidity tubes from entering the turbidimeter light chamber.

NEVER PUT WET TUBES IN THE TURBIDIMETER.

SAFETY PRECAUTIONS

Read the label on all reagent containers. Some labels include precautionary notices and first aid information. Certain reagents are considered hazardous substances and are designated with a * in the instruction manual. Material Safety Data Sheets (MSDS) are supplied for these reagents. Read accompanying MSDS before using these reagents. Additional emergency information for all LaMotte reagents is available 24 hours a day from the Poison Control Center listed in the front of the phone book. Be prepared to supply the name and four digit LaMotte code number found on the container label or at the top of the MSDS. LaMotte reagents are registered with a computerized poison control information system available to all local poison control centers.

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

Instrument Type	Nephelometric turbidity, calibrated in NTU
Range	0.00 -1100 NTU
Accuracy	.05 or ±2% for readings below 100 NTU, whichever is greater ±3% above 100 NTU
Resolution	*Standard Mode* 0.01 from 0.00 -10.99 NTU 0.1 from 11.0 -109.9 NTU 1 from 110 -1100 NTU *EPA Mode* NTU — Reported to the nearest NTU 0 - 1.0 — 0.05 1.0 - 10 — 0.1 10 - 40 — 1 40 - 100 — 5 100 - 400 — 10 400 - 1000 — 50 1000 — 100
Display	3½ digits
Response Time	5 seconds
Warm-up time	Not required
Automatic Shut Off	2 minutes
Lamp	Tungsten Filament bulb (approximate life 800 hours)
Sample	15 mL in capped tube
Sample Chamber	Accepts 25mm diameter flat-bottomed tubes (capped)
Power source	Battery Operation: 9 Volt Alkaline Line Operation: 120V/50Hz, 220V/60Hz*, with supplied adapter
Size (L X W X H)	8.5 x 16.2 x 6.7 cm, 3.4 X 6.4 X 2.6 inches
Shipping Weight	Meter only: 11 oz. (312g) Kit: 3 lb. 7 oz. (1560g)
Serial Interface	RS232, 8 pin mDIN, 9600b, 8, 1, n

**CE Mark: The devise complies to the product specifications for the Low Voltage Directive when furnished with the 220V AC Adapter (Code 1774). The 120V AC adapter is not CE approved.*

PARTS & ACCESSORIES

Included in the Model 2020 Turbidity Meter Kit (Code 1799 OR 1799-EX2):

Code	*Item*
26856	2020 Turbidity Meter
1726-110	AC Adapter, 9V (or 1726-220 with 1799-EX2)
1476	AMCO™ 2020 Turbidity Standard, 1.0 NTU, 60 mL
1477	AMCO™ 2020 Turbidity Standard, 10 NTU, 60 mL
0286-4	Turbidity tubes, set of 4

Optional Accessories:

1478	AMCO™ 2020 Turbidity Standard, 100 NTU, 60 mL
1479	AMCO™ 2020 Turbidity Standard, 250 NTU, 60 mL
1800	High Turbidity Dilution Kit includes: Syringe, Filter Holder, Membrane Filters
0943	Syringe
0598	Filter holder
1103-6	Membrane Filters, 0.45 micron, pkg of 6
5115PS-H	Deionized Water, 60 mL
6195-H	Formazin Turbidity Standard, 4000 NTU, 60 mL

EPA COMPLIANCE

This instrument meets or exceeds EPA design specifications for NPDWR and NPDES turbidity monitoring programs as specified by the USEPA method 180.1. There is also a compliance reading mode which rounds the reading to meet EPA reporting requirements.

WARRANTY

This instrument is guaranteed to be free from defects in material and workmanship for one year from original purchase date. If within that time the instrument is found to be defective, it will be repaired without charge except for transportation costs. The guarantee does not cover batteries.

CE COMPLIANCE

The 2020 Turbidimeter has been independently tested and has earned the European CE Mark of compliance for electromagnetic compatibility and safety.

DECLARATION OF CONFORMITY

Application of Council Directives:	89/336/EEC
Standards to which Conformity Declared:	EN55022, EN50082-1, En600950
Manufacturer's Name:	LaMotte Company
Manufacturer's Address:	802 Washington Avenue PO Box 329 Chestertown, MD 21620
Importer's Name:	Reagecon Diagnostics Ltd
Importer's Address:	13 A/D Shannon Free Zone Shannon, Co. Clase. Ireland
Type of Equipment:	Water Quality Meters
Model Number:	2020/1200
Year of Manufacture:	1997

I, the undersigned, hereby declare that the equipment specified above conforms to the above Directive and Standards.

Chestertown, Maryland
Place

James K. Trumbauer
Signature

3-19-97
Date

James K. Trumbauer
Name

V.P., Director of Research & Development
Position

NOTE: The device complies to the product specifications for the Low Voltage Directive when furnished with the 220V AC Adapter (Code 1774).

WHAT IS TURBIDITY?

Turbidity, cloudiness in water, can be interpreted as an absence of clarity or brilliance. It is caused by suspended and colloidal matter such as clay, silt, organic and inorganic matter and microscopic organisms. Turbidity should not be confused with color since a darkly colored water can still be clear and not turbid.

Turbid water is often an indicator of conditions that could cause damage to manufacturing equipment. Water clarity is especially important to the producers of consumer products such as beverage producers, food processors and water treatment plants. The particulates that cause turbidity may not always be harmful to human health, but are considered an undesirable characteristic.

Turbidity in industrial water used for boiler and cooling systems should be as low as possible. In boilers, the particles may become concentrated and settle out as a sludge that will damage equipment and cause foaming. In cooling water systems, particles can interfere with corrosion inhibitors. Water clarity is improved with fluid-particle separation processes such as sedimentation, coagulation and filtration.

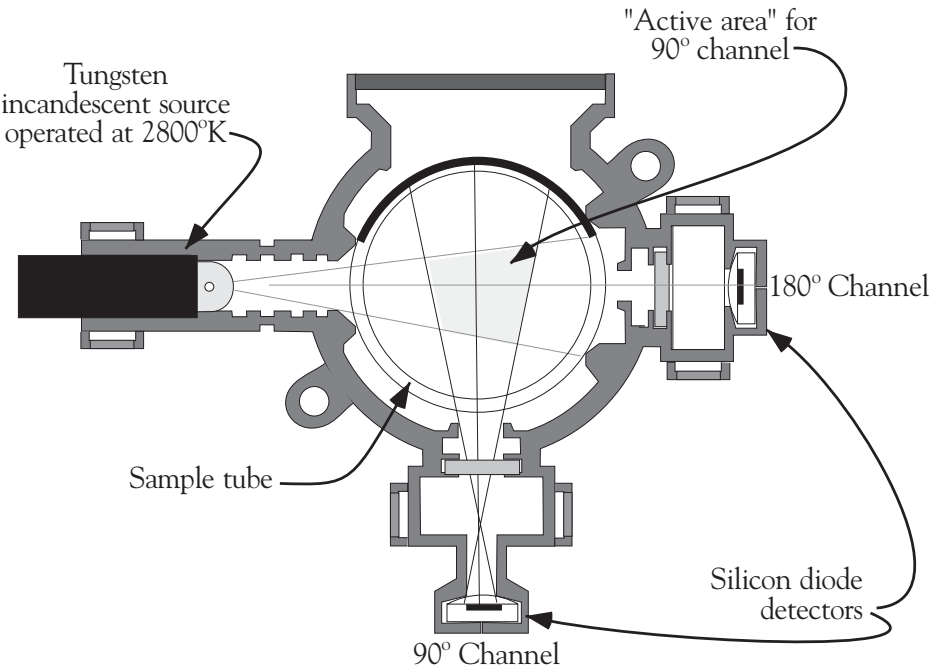
In swimming pools, cloudy water is a common problem. The usual causes for poor water clarity are corrosion, improper filtration and/or improperly balanced water. An algae condition or severe chloramine condition can also cloud pool water.

In natural waters, turbidity is an indicator of quality and productivity and can be used to monitor the health of streams and lakes. Turbid water may indicate runoff from construction, agriculture or other types of pollution. Suspended sediment can carry nutrients and pesticides throughout the water system. Suspended particles near the surface absorb additional heat from sunlight, raising the water temperature and blocking out the light needed by submerged aquatic vegetation and bottom dwelling creatures.

HOW IS TURBIDITY MEASURED?

Light passing through clear water will travel in a straight line. Particles in turbid water will cause the light to scatter giving it a "cloudy" appearance. The turbidity of a sample is determined by measuring the amount of scatter when a light is passed through a sample. The higher the turbidity, the greater the amount of scatter.

Turbidity can be measured in many ways. Visual methods include, the comparative methods, the Secchi disk method and the Jackson Candle method. Comparative methods are used in shallow water and determine turbidity by matching the turbidity of a water sample to a standard of known turbidity either with a "target" at the bottom of a tube or with a turbidity comparator. In the deeper waters of lakes, ponds, rivers and estuaries the Secchi disk is often used to measure turbidity. The Secchi disk is a disk about eight inches in diameter that is either white or is marked with black and white quadrants. The disk is lowered into the water on a calibrated line and the depth is noted where the disk just disappears from sight. The disk is then raised until it is visible. The average of these two distances is known as the "Secchi depth".



2020 Nephelometer

At waterworks and wastewater treatment plants the Jackson Candle apparatus was a standard instrument for measuring turbidities of incoming raw waters and treated wastewater effluents for many years. The equipment was modified over time but originally it consisted of a long glass tube supported over a "standard candle." Water was added to or removed from the tube until the image of the candle flame became indistinct. The depth of the water in the tube was read off a calibrated scale etched into the side of the tube, and results were reported numerically as Jackson Turbidity Units (JTU). The lowest turbidity that can be determined with this method is 25 Nephelometric Turbidity Units (NTU). Since the EPA's Surface Water Treatment requirements state that, finish water from municipal treatment plants will have a turbidity less than 1 NTU, indirect methods were developed to measure turbidity. Turbidimeters are the preferred method.

Nephelometers, such as the 2020, are turbidimeters that measure the scattered light at 90 degrees from the light source. A reference beam passes through the sample and is measured at 180 degrees. The ratio of these two readings is electronically converted to a turbidity measurement in NTU.

GENERAL OPERATING INFORMATION

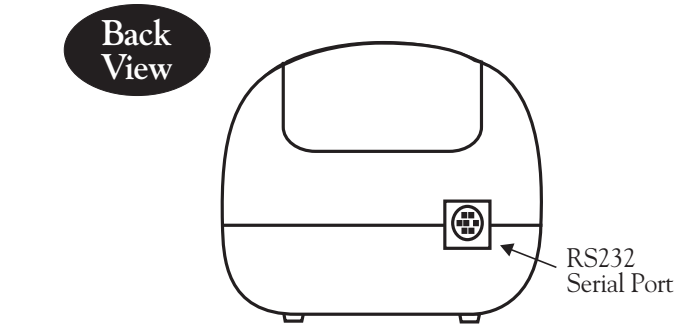
OVERVIEW

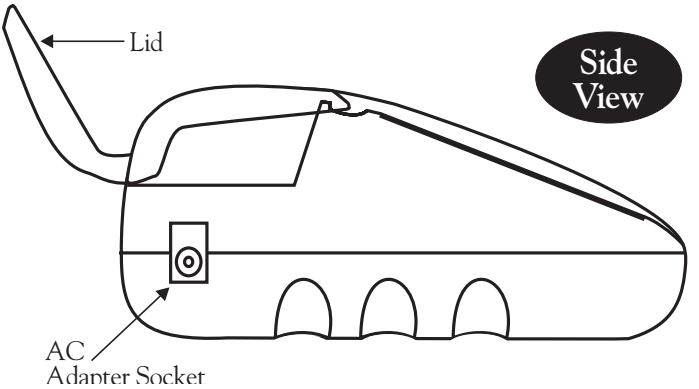
The 2020 Turbidimeter is a portable, microprocessor controlled nephelometer. A multi-detector optical configuration assures long term stability and minimizes stray light and color interferences. All readings are determined by the process of signal averaging over a 5 second period, minimizing fluctuations in readings attributed to large particles and enabling rapid, repeatable measurements. It has a sealed keypad. The microprocessor enables auto-ranging over the full range of 0 to 1100 NTU and provides direct digital readout with a resolution of 0.01 NTU for the lowest range.

The optics feature a tungsten bulb light source with a life expectancy of 800 hours. The light is detected by a silicon photo diode.

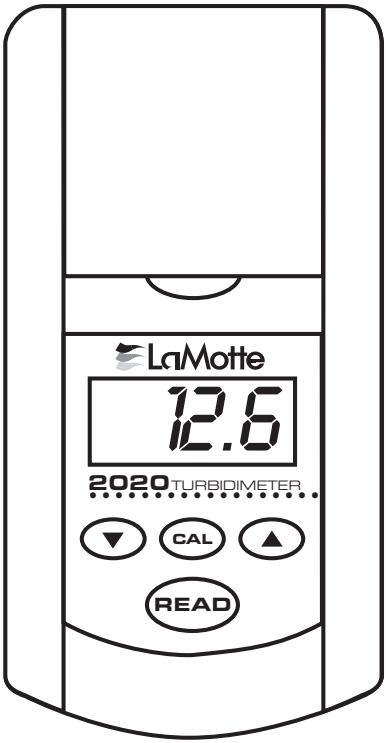
The 2020 is supplied with a 9 volt alkaline battery and an AC power adapter. A fresh battery should be installed at all times, even when using the power adapter. This will ensure that the meter will power down properly.

A RS-232 serial port on the back of the meter allows an interface of the turbidimeter with an IBM compatible computer for real time data acquisition and data storage using the PC. This port also allows an interface with a RS-232 serial printer.

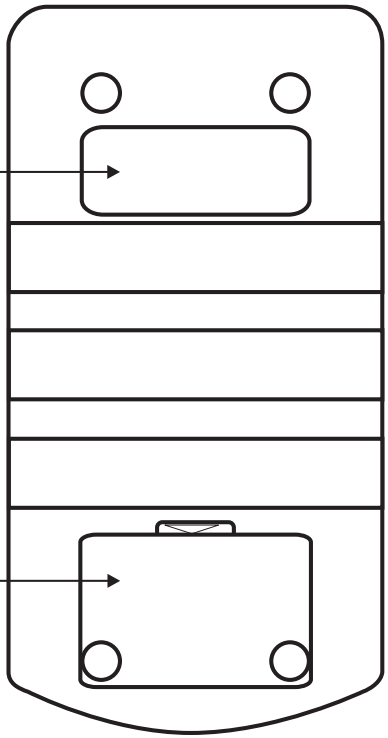




Top View



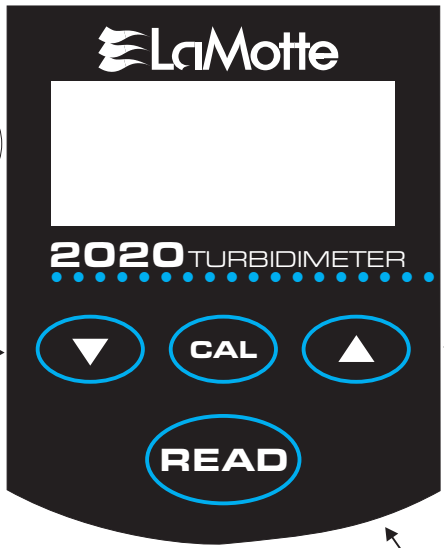
Bottom View



The DISPLAY will display turbidity reading with the following resolution:
0.00 - 10.99 NTU; 11.0 - 109.9 NTU; 110 - 1100 NTU

- When the **READ** button is first pushed, a number will be briefly displayed that indicates the software version number.
- A walking dash "-" will be displayed when measurement is taking place.
- The display will flash after the **CAL** button has been pushed during the calibration procedure until the **CAL** button has been pushed again to enter the adjusted value.
- "OFF" will be displayed after the **READ** button has been held down for 1 second. The meter will turn off when the button is released.
- "Er1" will be displayed when the battery voltage is very low.
- "Er2" will be displayed when measured turbidity is over range (1100 NTU).
- "Er3" will be displayed when the bulb has burned out or the tube is misaligned.
- "BAT" will be displayed when the battery voltage is getting low. Readings are reliable. Replace battery as soon as possible.
- "▲" will be displayed when the meter is in EPA mode.

See TROUBLE SHOOTING GUIDE page 23



The DOWN ARROW will **DECREASE** the numerical value of the display while in calibration mode.

The UP ARROW will **INCREASE** the numerical value of the display while in calibration mode.

The READ button is used to turn the meter ON and to take readings. Pressing the button for 1 second will cause the meter to display OFF. Releasing the button when OFF is displayed turns the meter OFF.

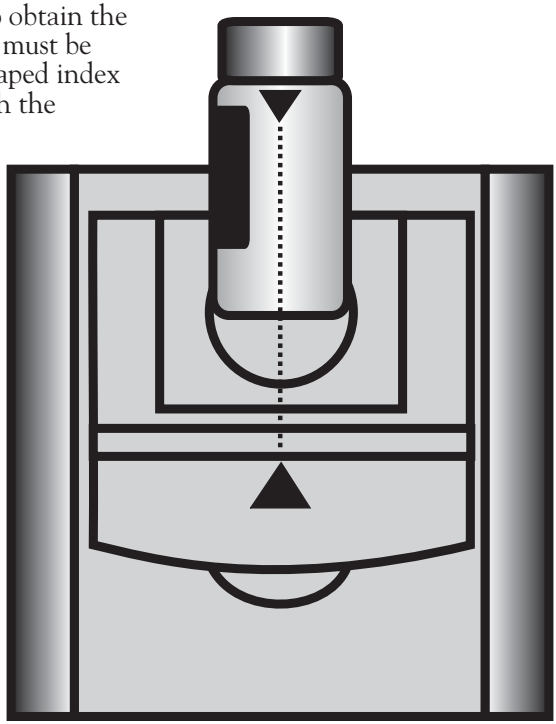
The CAL button is used for **CALIBRATION** procedures and to change between standard operating mode and EPA mode.

TURBIDITY TUBES

Turbidity tubes should always be washed prior to use. Use a mild detergent to remove any dirt or finger prints. Dry the outside of the turbidity tubes with a clean, lint-free cloth or disposable wipe. Allow the turbidity tubes to air-dry in an inverted position to prevent dust from entering the tube.

The handling of the turbidity tubes is of utmost importance. Scratches, fingerprints and water droplets on the turbidity tube or inside the light chamber can cause stray light interference leading to inaccurate results. It is imperative that the turbidity tubes and light chamber be clean and dry. Scratches and abrasions will permanently affect the accuracy of the readings. The inside of the tubes can be acid washed periodically and coated with special silicon oil to mask imperfections in the glass. Avoid acid contact with the black ink on the outside of the tubes. 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.

Variability in the geometry and quality of the glassware is the predominate cause of variability in results. The special anti-reflective area on the 2020 tubes allows more accurate turbidity readings for low NTU samples. Only 2020 tubes should be used with the 2020 turbidimeter. Orientation of the tube in the chamber will greatly affect the test results. To obtain the most accurate results, the tubes must be positioned so that the arrow-shaped index mark on the tube is aligned with the arrow-shaped index mark molded into the housing in front of the light chamber. This will ensure that the most accurate results are obtained.

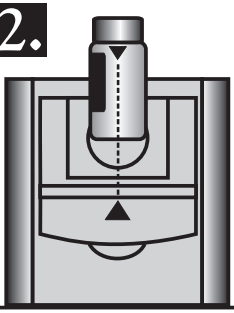


The 2020 turbidity tubes are optically selected but very small variations in the tubes may cause different readings on the same sample in low turbidity water. If greater accuracy is required, such as for Drinking Water requirements, the tubes supplied with the 2020 should be individually calibrated. This procedure is important for reading below 10 NTU but is probably not needed for samples above 10 NTU.

1.

Fill each tube (0286) with high-quality water. (Generally distilled water or drinking water is sufficient.)

2.

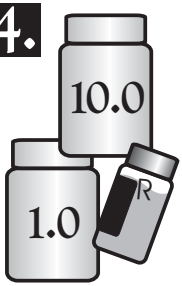


Record a turbidity reading for each tube following the instructions for turbidity measurement on page 19.

3.

Mark the tube with the lowest reading with an "R", for reference turbidity tube, near the top of the tube with a permanent marker.

4.

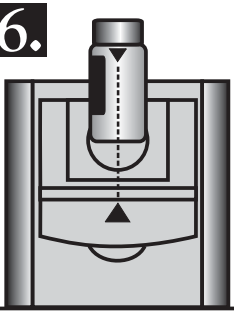


Follow the Calibration Procedure on page 17 using the reference turbidity tube, turbidity-free water, and the 1.00 NTU or 10.0 NTU AMCO™ standard.

5.

Fill the remaining turbidity tubes with the same AMCO™ turbidity standard as in the reference turbidity tube.

6.



Insert each tube into the meter with the proper orientation, Close lid and record the reading for each tube.

7.

The difference between the theoretical value (1.00 NTU or 10.0 NTU) and the actual value is the correction factor for that tube. This factor should be used when comparing results from different tubes.

CALIBRATION

STANDARD SOLUTIONS

The 2020 has been pre-calibrated in the range of 0 to 1100 NTU with AMCO™ primary standards manufactured by Advanced Polymer Systems, Inc. This allows the 2020 to be used for treated water, natural water or wastewater. Recalibration of the 2020 by the user is not required. However, a procedure to standardize the calibration should be performed to obtain the most accurate readings over a narrow range.

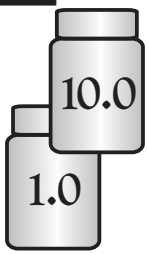
Two AMCO™ standards of 1.00 NTU and 10.0 NTU are supplied with the 2020. Standards of other values are available as accessories. The standards are a suspension of uniformly sized plastic "micro spheres" in ultra pure water, which require no preparation and are stable for long periods of time. These standards were manufactured specifically as a reference to calibrate the 2020. Only LaMotte specific AMCO™ standards should be used with the 2020. These standards are guaranteed to be accurate to within ±1%, if the following precautions are observed:

- The standards will remain stable for up to 4 years prior to opening if stored between 10 and 40°C.
- Once the seal of the bottle is broken, the stability of the standard is only guaranteed for 1 year if stored between 10 and 40°C.
- Never pour any unused or used standard back into the primary standard bottle.
- Do not open the bottle in a dusty or dirty environment. Dust and contaminants from the air can ruin the quality of the standard solutions.
- Before filling a tube with a standard, rinse the inside of the tube with a small amount of standard.
- Cap the standard bottle and the tube immediately after filling.

With proper preparation techniques, freshly prepared Formazin standards should be equivalent to the AMCO™ standards and can be used for meter calibration. A 4000 NTU Formazin Standard is available from LaMotte Company for use in preparing calibration standards. (See "Optional Accessories," pg. 6.) Correct procedures and approved methods for the use of Formazin standards can be found in the current edition of Standard Methods for Examination of Water and Wastewater.

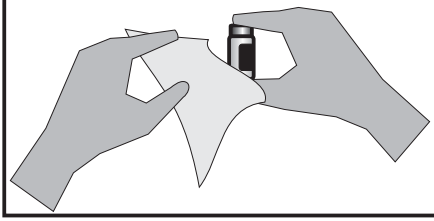
CALIBRATION PROCEDURE

1.

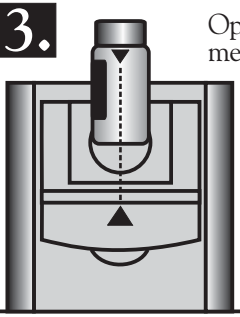


Select a LaMotte AMCO™ 2020 Standard in the range of the samples to be tested. NOTE: Only use LaMotte AMCO™ Standards specific to the 2020 Turbidimeterr. Contact LaMotte for replacement standards.

2. Fill a turbidity tube with the standard, cap, and wipe the tube clean with a lint-free cloth.

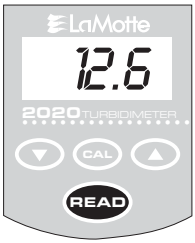


3.

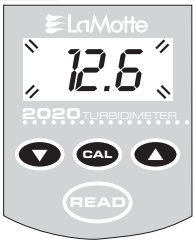


Open the lid of the meter. Align the indexing arrow mark on the tube with the indexing arrow mark on the meter, and insert the tube into the chamber.

4. Close the lid. Push the **READ** button. If the displayed value is not the same as the value of the reacted standard (within the specification limits), continue with the calibration procedure.

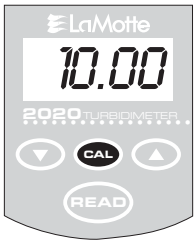


5.



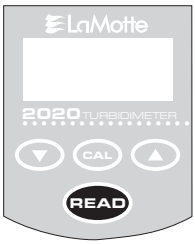
Push the **CAL** button for 5 seconds until **CAL** is displayed. Release button. The display will flash. Adjust the display with the ▼ and ▲ buttons until the value of the standard is displayed.

6.



Push the **CAL** button again to memorize the calibration. The 2020 display will stop flashing. Calibration is complete.

7.



Turn the unit off by holding the **READ** button down for at least 1 second, or proceed to measure the test samples following the procedure on page 19.

Note

The calibration procedure should be followed once a week, or more often as required by regulations and laws for compliance monitoring. The calibration of the meter is independent of the operating mode.

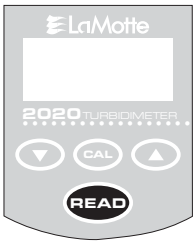
ANALYSIS PROCEDURES

SELECTING THE EPA MODE

The 2020 turbidity meter has two operating modes, the standard operating mode and the EPA mode. The meter can only be switched from one mode to the other while turning the 2020 on, from the OFF state. The 2020 will remain in which ever mode it was last used, even if the meter has been turned OFF.

To switch from one mode to the other mode:

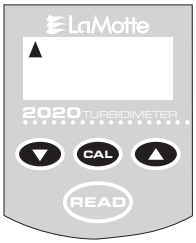
1.



Turn OFF the 2020, if it is on.

2.

Press **CAL** button and hold it down while pressing the **READ** button to turn the meter on.



The meter will come on in the opposite mode than it was in previously. (While in EPA mode the ▲ will be visible on the display).

The standard operating mode displays the measured turbidity to the full resolution of the meter. The EPA mode displays the measured turbidity rounded to the reporting requirements of the EPA and Standard Methods compliance monitoring programs. This greatly simplifies the reporting requirements by eliminating the need for the user to manually round off the results according to EPA specifications. The EPA requires these reporting requirements because it recognizes the inherent accuracy of turbidity measurements within the specified ranges.

Note: The calibration of the meter is independent of the operating mode.

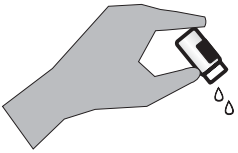
TURBIDITY MEASUREMENT

1.

Fill a clean container with at least 50 mL of sample water and cover. Set sample aside to allow sample to equilibrate to air temperature and let gases escape. Avoid contaminants. Analyze as soon as possible.

2.

Rinse an empty turbidity tube with a portion of the sample. Shake out excess water.

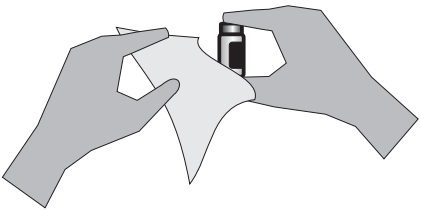


3.

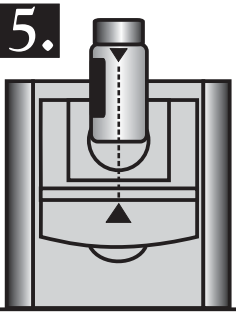
Fill the turbidity tube (0286) to the neck by carefully pouring the sample down the side of the tube to avoid creating bubbles.

4.

Cap the tube and wipe tube dry with a clean lint-free tissue.

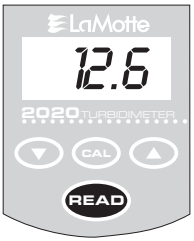


5.



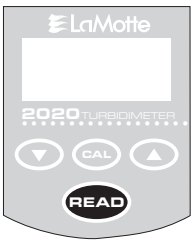
Open the 2020 lid. Align the indexing arrow on the tube with the indexing arrow on the meter. Insert the turbidity tube into chamber.

6.



Close the lid. Push the **READ** button. The turbidity in NTU units will be displayed within 5 seconds.

7.



The 2020 will turn off automatically 2 minutes after the last button push. To turn the meter OFF manually, hold the **READ** button down for at least 1 second. Release the button when OFF is displayed.

Note

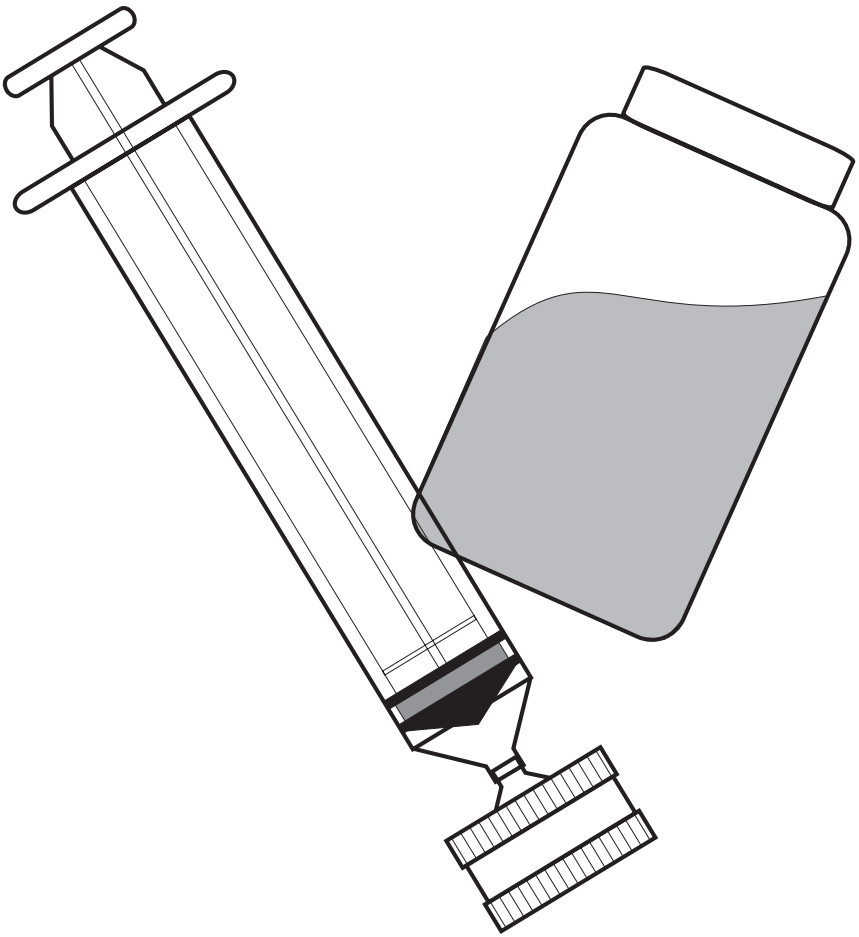
If the sample is higher than 1100 NTU, it must be diluted and retested. See pages 20-22.

PREPARATION OF TURBIDITY FREE WATER

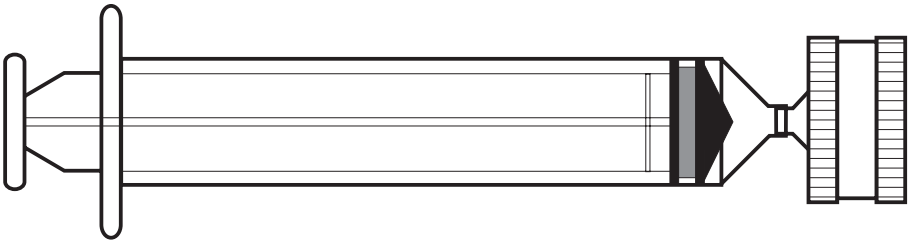
An accessory package (Code 1800, not included) is available for preparing turbidity free water for dilution of high turbidity samples.

The preparation of turbidity free water requires careful technique. Introduction of any 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 below 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.

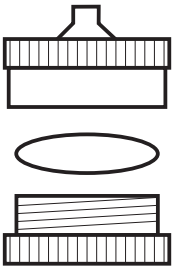
See procedure on next page…



PROCEDURE:



1.

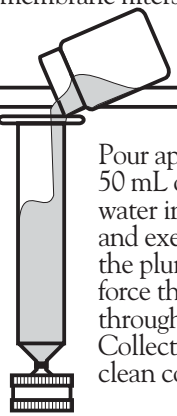


Unscrew the top of the filter holder (0598). (The black O-Ring should remain in the top part of the filter holder). Place a white membrane filter (1103) on the screen inside. Position the filter disk so that it covers the entire surface of the screen. Replace the top of the filter holder and screw on securely. Note: the membrane filters are white and packaged between two blue protective disks. Handle membrane filters with extreme care.

2.

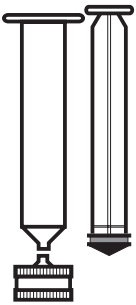
Remove the plunger from the syringe (0943). Attach filter holder to the bottom of the syringe.

3.



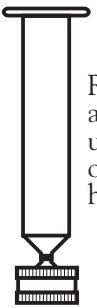
Pour approximately 50 mL of deionized water into the barrel and exert pressure on the plunger to slowly force the water through the filter. Collect water in a clean container.

4.



Remove the filter holder from the syringe, then remove the plunger from the barrel. (This step is required to prevent rupturing the membrane filter by the vacuum as the plunger is removed.)

5.



Replace the filter holder and repeat steps 3 and 4 until the desired amount of turbidity-free water has been collected.

Periodically examine the membrane filter to insure that no holes or cracks are present. Depending on the nature of the unfiltered water, it is possible to prepare a liter or more of turbidity-free water using a single filter. The membrane filter may be stored in the holder indefinitely and used as required.

DILUTION PROCEDURES

If a sample is encountered that is higher than 1100 NTU, a careful dilution will bring the sample into the acceptable range. However, there is no guarantee that halving the concentration will exactly halve the NTU values. The particulates often react in an unpredictable manner when diluted.

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. Discard tubes that are badly scratched.
4. Gently mix sample by inverting before taking a reading but avoid introducing air bubbles.
5. Turbidity readings will be affected by electric fields around motors.
6. Carbon in the sample will absorb light and cause low readings.
7. Observe shelf life recommendations for turbidity standards.
8. The turbidimeter should be placed on a surface free from vibration. Vibrations can cause high readings.
9. Excessive color in a sample will absorb light and cause high readings. The user should verify if a certain level of color will cause a significant error at the level of turbidity being tested.

TROUBLESHOOTING

PROBLEM	CHECK	ACTION
No Power	Battery	Replace
	AC Adapter	Plug in
	AC Wall Outlet	Verify power source
	Contact LaMotte for Return Authorization	Return to LaMotte for repair
Suspect Calibration	Check calibration with standards	Use new standards
	Verify standards with Formazin	Run alternate test with Formazin
	Verify with another meter	Check other meter calibrations
	Check tube alignment	Re-align tube
	Check sample tubes for dirt and scratches	Check, clean and/or replace if necessary
	Check to see if internal meter components are wet	Always dry tubes before inserting. Examine chamber for visible moisture.
	Reset meter to factory calibration	With meter off, hold down ▼ and press **READ**
	Battery	A fresh battery should be installed at all times, even when using the power adapter. This will ensure that the meter will power down properly and the calibration will not be lost. Return meter for recalibration.
	Contact LaMotte for Return Authorization	Return for calibration check
ER1	Very low battery	Change battery
ER2	Over range	Dilute sample
ER3	Burnt out bulb or misaligned tube	Check alignment Call LaMotte
BAT	Low Battery	Change battery

RS232 PORT

The 2020 Turbidimeter may be interfaced with any IBM compatible computer using an Interface cable (Code 1772). The meter may also be interfaced with an RS-232 serial printer, using an appropriate cable and setting the printer configuration to the output below.

Output: RS232 compatible, asynchronous serial, 9600 baud, no parity, 8 data bits, 1 stop bit.

Computer Connection: RS232 interface connection, 8 pin mDIN/9 pin F D-submin.

Pin out:

5	RS-232 TxD
3	RS-232 RxD
4, 6, 8	digital ground

MAINTENANCE

REPLACING THE BATTERY

The LaMotte 2020 uses a standard 9-volt alkaline battery that is available worldwide. A fresh battery should be installed at all times, even when using the power adapter. This will ensure that the meter will power down properly. The battery compartment is located on the bottom of the case. To replace the battery:

1. Open the battery compartment lid
2. Remove the battery and disconnect the battery from the polarized plug.
3. Carefully connect the new battery to the polarized plug and insert it into the compartment.
4. Close the battery compartment lid

REPLACING THE LAMP

The tungsten lamp included with the model 2020 has a life of approximately 800 hours. If the display becomes unstable when using LaMotte AMCO™ standards, call LaMotte Company for a return authorization number to have the lamp replaced and have the unit examined.

REPAIRS

If it is necessary to return the instrument for repair, telephone LaMotte Company at 1-800-344-3100 or fax 1-410-778-6394 for a return authorization number.

LaMOTTE COMPANY

Helping People Solve Analytical Challenges®

PO Box 329 • Chestertown • Maryland • 21620 • USA
800-344-3100 • 410-778-3100 (Outside U.S.A.) • Fax 410-778-6394
Visit us on the web at www.lamotte.com

10/02 • 61799