01326

Seneca Army Depot Activity
Romulus, New York

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

Addendum 1 to the Final UFP-QAPP

Seneca Army Depot Activity

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

October 2018

Table of Contents

LIST OF FIGURES

LIST OF TABLES

LIST OF APPENDICES

LIST OF ACRONYMS

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

Executive Summary

ES.1 Introduction

This Addendum to the existing Final UFP-QAPP (Parsons, 2017) covers the addition of perchlorate and 1,4-dioxane analysis at two sites and updates to sampling and laboratory procedures for additional poly- and perfluoroalkyl substances (PFAS) sampling. Additions included in this amendment to the UFP-QAPP include:

1) Perchlorate will be analyzed in soil and groundwater samples collected at the Open Detonation Grounds (OD Grounds). The historic use of the site was to detonate munitions and other ordinance. This site was not covered in the Final QAPP and therefore additions to the Addendum include site specific information regarding the OD Grounds, the perchlorate sampling approach, and applicable analytical and laboratory procedures.

2) Ash Landfill was included in the Final QAPP as part of the LTM program. Site specific details for the Ash Landfill are included in the Final QAPP. Per request by the NYSDEC, additional groundwater samples will be collected and analyzed for 1,4-dioxane at the Ash Landfill, where contaminants may be associated with chlorinated solvents in the groundwater. Additional details regarding the sampling methods and analytical details for 1,4-dioxane are included in this Addendum.

3) Based on the findings of the PFAS Site Inspection (Parsons, 2018), additional PFAS sampling was requested by the NYSDEC for the SEAD 25 and SEAD 26 sites. Site specific information for SEAD 25 and SEAD 26 is included in the Final QAPP. This Addendum includes updates regarding the scope of the PFAS sampling, sampling SOPs, analytical specifications and laboratory SOPs that may have changed since the Final QAPP was issued.

Information related to the other field programs already provided in the existing Final UFP-QAPP was removed for clarity. A brief overview of any additional sites to be investigated or changes in scope is provided below. A copy of the Final QAPP is included in **Appendix C**. This Addendum is intended to be used with the Final QAPP; the Addendum is not a stand-alone document.

ES.1.1 OD GROUNDS

The OD Grounds Site is located in the northwestern corner of the SEDA and was used to perform open detonation of munitions. The Site is largely meadow with some wooded and heavily brushed areas. The detonation activities at the OD Grounds were conducted in an area known as the "OD Hill." The historic operations have resulted in munitions and explosives of concern (MEC) and munitions debris (MD) being "kicked out" from the OD Hill to the surrounding area. Several munitions response actions were conducted within the OD Grounds since the year 2000. Future munitions related cleanup is expected at this Site. This QAPP Addendum addresses the addition of perchlorate sampling in soil and groundwater at the OD Grounds.

ES.1.2 ASH LANDFILL

The Ash Landfill, located in the west-central portion of SEDA, was used from 1941 to 1974 to burn uncontaminated trash in a series of burn pits located near the former abandoned incinerator building (Building 2207). Long term monitoring activities have been conducted since 2007 at the Ash Landfill to assess groundwater conditions related to a chlorinated solvent plume, the effectiveness of the biowall, and the vegetative soil cap. Additional site-specific information is included in ES.1.3 in the Final QAPP. Based on request from the NYSDEC, 1,4-dioxane will be sampled in a subset of the wells located at the Ash Landfill.

ES.1.3 SEAD 25 AND SEAD 26

SEADs 25 and 26 were former fire training areas. Additional site-specific detail is available in Sections ES.1.2 and ES.1.5 in the Final QAPP. In 2017, these two sites were part of a Site Investigation for PFAS compounds. Additional investigation of PFAS compounds will take place at these sites. Included in this Addendum are the proposed sampling scope and any updates to laboratory detection limits and SOPs.

ES.2 Project Objectives and Technical Approach

The project objectives are to investigate several emerging contaminates, by request of the NYSDEC and EPA, at sites within the SEDA. Additional PFAS groundwater sampling will be conducted at SEADs 25 and 26, perchlorate will be sampled in soil and groundwater at the OD Grounds, and 1,4-dioxane will be sampled in groundwater at the Ash Landfill. The Conceptual Site Model (CSM) for each site is described on **Worksheet #10**.

Project-specific data quality objectives (DQOs) were developed based on this CSM and these are described on **Worksheet #11** of this UFP-QAPP Addendum. These DQOs include a design for obtaining data to support the sampling of emerging contaminants including PFAS, perchlorate and 1,4-dioxane. The design for obtaining data described in the last column of the DQO tables on **Worksheet #11** summarizes the technical approach. The project approach is described in detail on **Worksheet #17**, and specific analyzes are noted on **Worksheet #18**. The primary components of the sampling design for each of the sites involve collecting a limited number of soil samples and groundwater samples to be analyzed using low-flow techniques.

The general scope of the activities related to sampling for perchlorates at the selected site is as follows:

- **OD Grounds**
  - Collect groundwater samples from groundwater wells
  - Collect soil samples at two depths; and
  - Analyze samples for perchlorate.

The general scope of the activities related to sampling for 1,4-dioxane at the Ash Landfill include:

- **Ash Landfill**
  - Collect groundwater samples using a subset of the existing wells at the Ash Landfill; and
  - Analyze samples for 1,4-dioxane.

The general scope of the activities related to sampling for PFAS at SEADs 25 and 26 include:

- **SEAD 25 and SEAD 26**
  - Install new monitoring wells at the firehouse north of SEAD 25, around the perimeter of SEAD 25, and at locations within and downgradient of SEAD 26;
  - Collect one round of groundwater samples; and
  - Analyze samples for PFAS compounds.

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

ES.3 Document Organization

This UFP-QAPP Addendum was prepared under Task Order 0023 of Contract W912DY-09-D-0062, in accordance with UFP-QAPP, Optimized UFP-QAPP Worksheets (EPA, 2012), EPA QA/G-5 (EPA, 2002), and EM 200-1-15 to ensure environmental data collected are scientifically sound, of known and documented quality, and suitable for their intended

purposes. This UFP-QAPP Addendum focuses on the site-specific details for the additional PFAS sampling at SEADs 25 and 26, perchlorate sampling at the OD Grounds and 1,4-dioxane sampling at the Ash Landfill. The site-specific details include monitoring methods, analytical services, data management and validation procedures, and field and laboratory SOPs. This Addendum is intended to be reviewed and used with the Final UFP-QAPP.

This UFP-QAPP addendum uses the "optimized" worksheets format published by the Intergovernmental Data Quality Task Force in March 2012 (EPA, 2012). Supporting plans and other information are included in the references section of this UFP-QAPP.

Crosswalk from UFP-QAPP Manual to Worksheets

This UFP-QAPP Addendum presents the plan for collecting data to support additional PFAS sampling at SEADs 25 and 26, perchlorate sampling at OD Grounds and 1,4-dioxane sampling at the Ash Landfill. The UFP-QAPP uses "optimized" worksheets published by the Intergovernmental Data Quality Task Force in March 2012. The optimized worksheets address all requirements of ANSI/ASQ E4-2004 and CIO 2106-G-05. The following table provides a "crosswalk" between the worksheets and the respective elements of CIO 2106-G-05. In addition, each revised worksheet includes a reference to the appropriate CIO 2106-G-05 element. Worksheets without changes were removed and are noted in the table below. These worksheets may be referenced in the Final UFP-QAPP (Parsons, 2017).

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

OPTIMIZED UFP-QAPP WORKSHEETS		2106-G-05 QAPP GUIDANCE SECTION	
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection	2.3.6	Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables
26 & 27	Sample Handling, Custody, and Disposal	2.3.3	Sample Handling, Custody Procedures, and Documentation
28	Analytical Quality Control and Corrective Action	2.3.5	Quality Control Requirements
29	Project Documents and Records	2.2.8	Documentation and Records Requirements
31, 32 & 33	Assessments and Corrective Action (removed, refer to Final UFP-QAPP)	2.4 2.5.5	Assessments and Data Review Reports to Management
34	Data Verification and Validation Inputs (removed, refer to Final UFP-QAPP)	2.5.1	Data Verification and Validation Targets and Methods
35	Data Verification Procedures (removed, refer to Final UFP-QAPP)	2.5.1	Data Verification and Validation Targets and Methods
36	Data Validation Procedures	2.5.1	Data Verification and Validation Targets and Methods
37	Usability Assessment (removed, refer to Final UFP-QAPP)	2.5.2 2.5.3 2.5.4	Quantitative and Qualitative Evaluations of Usability Potential Limitations on Data Interpretation Reconciliation with Project Requirements

Worksheets #1 & 2: Title and Approval Page

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

1.1 PROJECT IDENTIFYING INFORMATION

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

1.2 CONCURRING SIGNATURES

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

Lead Organization / Contracting Officer Representative	Derek Pommerenck	25Oct2018
	Derek Pommerenck, USACE CEHNC MMDC Point of Contract/COR Project Manager	Date
Lead Organization / Project Manager	RWBattaglia	26 Oct 2018
	Randy Battaglia, USACE New York District (CENAN)	Date
Contractor Project Manager	Beth Badik	10/19/18
	Beth Badik, Parsons Project Manager	Date
Federal Regulatory Agency		
	Bob Morse, USEPA Regional Project Manager	Date
State Regulatory Agency		
	Melissa Sweet, NYSDEC Project Manager	Date
Contractor Quality Assurance	Beth Driskill	10/29/18
	Beth Driskill, Parsons Quality Manager	Date

1.3 QAPP IDENTIFYING INFORMATION

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

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

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

4.1 KEY PROJECT PERSONNEL

PROJECT TITLE/ROLE	NAME/ ORGANIZATION	CONTACT INFORMATION (TELEPHONE/E-MAIL)	EXPERIENCE	SPECIALIZED TRAINING/ CERTIFICATIONS	SIGNATURE/DATE [1]
USACE Contracting Officer Representative (COR)	Derek Pommerenck USAESCH	256-895-1794 Derek.Pommerenck@usace.army.mil	n/a	n/a	*Signature on Worksheets #1 & 2*
USACE Project Manager (PM)	Randy Battaglia CENAN	347-213-1565 Randy.W.Battaglia@usace.army.mil	n/a	n/a	*Signature on Worksheets #1 & 2*
Contractor PM	Beth Badik Parsons	617-449-1565 beth.badik@parsons.com	Over 10 years of experience as PM conducting HTRW investigations	BS, Chemical Engineering, 2001	*Signature on Worksheets #1 & 2*
Federal Regulator	Bob Morse USEPA Region 2	212-637-4331 Morse.Bob @epa.gov	n/a	n/a	*Signature on Worksheets #1 & 2*
State Regulator	Melissa Sweet NYSDEC	518-402-9614 melissa.sweet@dec.ny.gov	n/a	n/a	*Signature on Worksheets #1 & 2*

4.2 OTHER PROJECT PERSONNEL

PROJECT TITLE/ROLE	NAME/ ORGANIZATION	CONTACT INFORMATION (TELEPHONE/E-MAIL)	EXPERIENCE	SPECIALIZED TRAINING/ CERTIFICATIONS [1]	RECEIVES COPY OF QAPP
Analytical Laboratory Project Manager	Linda C. Laver TestAmerica- W. Sacramento	916-374-4362 linda.laver@testamericainc.com	Over 18 years of Project Management, 2 years in environmental consulting, and 5 years in analytical lab	B.S Resource Sciences, 1985	No
Analytical Laboratory QA Officer	Roxanne Sullivan TestAmerica Laboratories-Denver	303-736-0116 Roxanne.Sullivan@testamericainc.com	Over 30 years of analytical laboratory and chemistry-related experience	B.S., Chemistry	Yes
Analytical Laboratory Laboratory Director	Richard Clinkscales TestAmerica Laboratories-Denver	303-828-8811 Richard.Clinkscales@testamericainc.com	Over 30 years of analytical laboratory and chemistry-related experience	B.S., Chemistry and English	Yes

Worksheet #9: Project Planning Session Summary

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

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

NAME	ORGANIZATION	TITLE / ROLE	E-MAIL / PHONE
Randy Battaglia	CENAN	Seneca AD BRAC Environmental Coordinator/Caretaker	Randy.W.Battaglia@usace.army.mil 347-213-1565 (o)
Derek Pommerenck	CEHNC	Project Manager	Derek.Pommerenck@usace.army.mil 256-374-6251 (c)
Barry Hodges	CEHNC	Technical Manager	Barry.A.Hodges@usace.army.mil 256-895-1894 (o) /256-503-0153 (c)
Mike D'Auben	CEHNC	Chemist	Michael.J.D'Auben@usace.army.mil 256-895-1460 (o)
Beth Badik	Parsons	Project Manager	Beth.Badik@parsons.com 617-449-1565 (o)
Todd Belanger	Parsons	Deputy Project Manager	Todd.Belanger@parsons.com 617-449-1428 (o)

Worksheet #10: Conceptual Site Model

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

10.1 OVERVIEW

The primary purpose of this worksheet is to describe the CSMs for each of the project sites. In order to provide the basis for this, this worksheet also summarizes observations from previous investigations, information from site reports, on-going LTM, details of the contaminants and the affected matrices, and other relevant supporting information. Further details for each site are available in the respective LTM reports. The Ash Landfill, SEAD 25 and SEAD 26 site were previously described in the Final QAPP. Additional information was added regarding the PFAS site investigation results at SEAD 25 and SEAD 26.

10.1.1 SITE LOCATION

OD Grounds

The OD Grounds Site is located in the northwestern corner of the Depot in Seneca County, New York and is also known as SEAD 006-R-01 (formerly SEAD-45 and SEAD-115). The Site is largely meadow with some wooded and heavily brushed areas. The OD Grounds was used to perform open detonation of munitions. The OD Grounds retained parcel consists of 403 acres. As defined in historical studies for the use of assigning work areas, the OD Grounds was assigned a series of radii (500ft, 1,000ft, 1,500ft, 2,000ft, and 2,500ft) centered on the OD Hill. The general area (421 acres) of greatest impact is defined by the 2,500-foot radius. This acreage includes the area surrounded by a 2,500-foot radius centered around the OD Hill; however, this radius does not define the limit of potential contamination. Note that the Open Burning (OB) Grounds (also known as SEAD-23; 30.2 acres) is a separate site that was previously addressed and is not included in the calculation of the OD Grounds acreage. For ease of discussion, two different portions of the OD Grounds Site were identified. They are referred to as the “Kickout Area” and the “OD Hill Area”. The OD Hill Area is the location of demolition activities. The Kickout Area is the area in which blast fragments emanating from the OD Hill activity are expected to land.

Access into the greater OD Grounds demolition area is possible via a paved road that enters the area from the southeast and roughly parallels the path of Reeder Creek along its western bank. The unnamed access road branches off North-South Baseline Road near Building 2104, which is located in the southeastern corner of the OD Grounds. Building 2104 was built in 1951 and is described as “Change House (OB/OD Grounds)”. The building is not included in any lists of structures with potential unexploded ordnance (UXO) hazards or in which potentially hazardous materials were stored (Woodward-Clyde, 1997). A change house is a location for military personnel to change clothes and uniforms.

10.2 HISTORY

10.2.1 OD GROUNDS

The OD Grounds was used to destroy munitions. Operations at the OD Grounds began circa 1941 when the Depot was first constructed and continued at regular intervals until circa 2000 when the military mission of the Depot ceased. Detonations occurred intermittently since the Depot closed as part of continuing munitions response activities being performed at the Depot. During operations, waste munitions are placed in a hole created in the hill with additional demolition material, covered with a minimum of 8 feet of soil, and detonated remotely. After demolition was completed, explosively displaced portions of the mound were reconstructed by bulldozing displaced and native soils back into the central earthen mound.

10.2.2 OD GROUNDS

The OD Grounds was used from 1941 to 2000 for operations related to the mission of the SEDA. The planned future use for OD Grounds is for conservation and passive recreational purposes where there is a limited potential for soil contact.

10.3 PREVIOUS INVESTIGATIONS

10.3.1 OD GROUNDS

Several characterization efforts and investigations for MPPEH and MC were conducted at the OD Grounds. No previous investigations have sampled for perchlorate at this site. A summary of the characterization and investigation efforts is presented in the following table:

PREVIOUS INVESTIGATION	YEAR	SUMMARY
ESI (Engineering Science, Inc., 1995)	1993-1994	Geophysics, test pitting, groundwater and surface water sampling conducted.
Archives Search Report (USACE, 1998)	1998	Site inspection, archives search and employee interviews to document previous military use and potential environmental contamination that could remain at the Seneca Army Depot.
OE EE/CA (Parsons ES, 2004)	2000	Characterized the nature and extent of MEC at the OD Ground using geophysical survey techniques and intrusive investigations.
Phase I Geophysical Investigation (Weston, 2005)	2003	Geophysical surveys collected using EM61 MK2 towed-array system to identify 14,700 anomalies within open areas between the 1,000 ft. and 1,500 ft. radius of OD Hill.
Phase II OE Removal Activities (Weston, 2006)	2003-2005	Reacquired, removed, and disposed of approximately 8,500 MEC/UXO and MD items located between the 1,500 ft. and 2,500 ft. radius from the OD Hill to a depth of 4 ft.
Additional Munitions Response Site Investigation (Parsons, 2010)	2010	Topographic and geophysical surveys of portions of the OD Grounds and the collection and analysis of soil samples from test pits (TP) and surface locations.
Munitions Response Action (Parsons, 2016)	2012-2014	Reacquired, and investigated 14,688 anomalies; used analog methods to remove UXO/DMM, and dispose of 15,885 munitions related items located between the 1,500 ft. and 2,500 ft. radius from the OD Hill to a depth of 4 ft.
MEC Clearance at OD Grounds	2012	Prior to early termination of contract, DGM survey of inner 1,000 feet completed.
Perchlorate Sampling	2018	Perchlorate sampling in soil, groundwater, ditch soil, and surface water.

10.3.2 SEAD 25 AND SEAD 26

During the Site Inspection, the two primary PFAS constituents, PFOA and PFOS, were detected at all three sites investigated at SEDA. Exceedances of the EPA Health Advisory for combined concentrations of PFOS/PFOA were observed at SEAD 25 and SEAD 26, but there were no exceedances observed at SEAD 122E. These data suggest the potential use of AFFF at SEADs 25 and 26, but not at SEAD 122E (Parsons, 2018b).

SEAD 25

- PFOA and PFOS were detected in 12 of 12 wells.
- The combined PFOA/PFOS concentration exceeded the EPA advisory health level in all 12 wells sampled.
- PFOA (alone) exceeded the EPA health advisory level in two wells.

- Detections of PFAS components, with elevated concentrations of PFOA and PFOS, indicate the potential use of AFFF or similar material at SEAD 25.

SEAD 26

- PFOA and PFOS were detected in 8 of 8 wells analyzed.
- Combined PFOA/PFOS exceeded the EPA health advisory level in four wells. Three of these locations are downgradient of the former fire training pit.
- PFOA (alone) exceeded the EPA health advisory level in four wells; three wells located downgradient of the former fire training pit and one well located upgradient, but in close proximity, to the pit.
- Four wells had no exceedance above the combined PFOA / PFOS advisory concentration.
- Detections of PFAS components, with elevated concentrations of PFOA and PFOS, indicate the potential use of AFFF or similar material at SEAD 26.

10.4 CONCEPTUAL SITE MODEL

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

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

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

A CSM was developed for each site for which additional analytes will be sampled. The CSMs for the Ash Landfill, OD Grounds, SEAD 25 and SEAD 26 are summarized in **Tables 10.1, 10.2, 10.3, and 10.4.** These tables describe the known or suspected contamination sources, potential/suspected location and distribution of contamination, contamination source or exposure medium, current and future receptors, and potentially complete exposure pathways. The CSM will be revised based on investigation results and Army and stakeholder feedback.

Except for at the OD Grounds, surface and subsurface soil pathways are incomplete at the Ash Landfill because the source was removal and buried under a soil cover. Surface and subsurface soil pathways are complete at the OD Grounds because the source was never removed. Surface water and sediment pathways are incomplete at SEADs 25 and 26 because the media do not exist. Currently, surface water and sediment pathways are incomplete at the OD Grounds because of creek monitoring conducted during LTM at the OB Grounds which shows no evidence of erosion of on-site soils to the creek. Additionally, no surface water or sediment are present within the OD Hill area. Exposure to groundwater through ingestion is considered potentially complete at Ash Landfill, SEAD 25, SEAD 26 and OD Grounds for current and future on-site workers, current and future off-site residents, and future residents if groundwater is accessed.

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

Table 10.1 - Overview of Preliminary Conceptual Site Model, Ash Landfill, Seneca Army Depot Activity

SITE DETAILS	KNOWN OR SUSPECTED CONTAMINATION SOURCE(S)	POTENTIAL/SUSPECTED LOCATION AND DISTRIBUTION	SOURCE OR EXPOSURE MEDIUM	CURRENT AND FUTURE RECEPTORS	POTENTIALLY COMPLETE EXPOSURE PATHWAY
NAME: Ash Landfill **Acreage**: 45 acres **Release mechanisms**: Former use of incinerator and burial of ash. **Current and Future Land Use**: No current use. Future use is planned industrial development.	VOCs (predominantly chlorinated VOCs); 1,4-dioxane	Potentially present in groundwater. Former soil source was removed prior to LTM.	Groundwater	Current and future onsite worker, off-site residential. Future on-site residential.	Exposure to groundwater (ingestion)

Table 10.2 - Overview of Preliminary Conceptual Site Model, OD Grounds, Seneca Army Depot Activity

SITE DETAILS	KNOWN OR SUSPECTED CONTAMINATION SOURCE(S)	POTENTIAL/SUSPECTED LOCATION AND DISTRIBUTION	SOURCE OR EXPOSURE MEDIUM	CURRENT AND FUTURE RECEPTORS	POTENTIALLY COMPLETE EXPOSURE PATHWAY
NAME: OD Grounds **Acreage**: 421 acres **Release mechanisms**: Demilitarization of munitions was performed via open detonation on the ground surface **Current and Future Land Use**: No current use. Proposed future use is conservation/recreation.	Perchlorate	Potentially present in groundwater as a result of leaching of the soil.	Groundwater	Current and future onsite worker, off-site residential. Future on-site residential.	Exposure to soil Exposure to groundwater (ingestion)

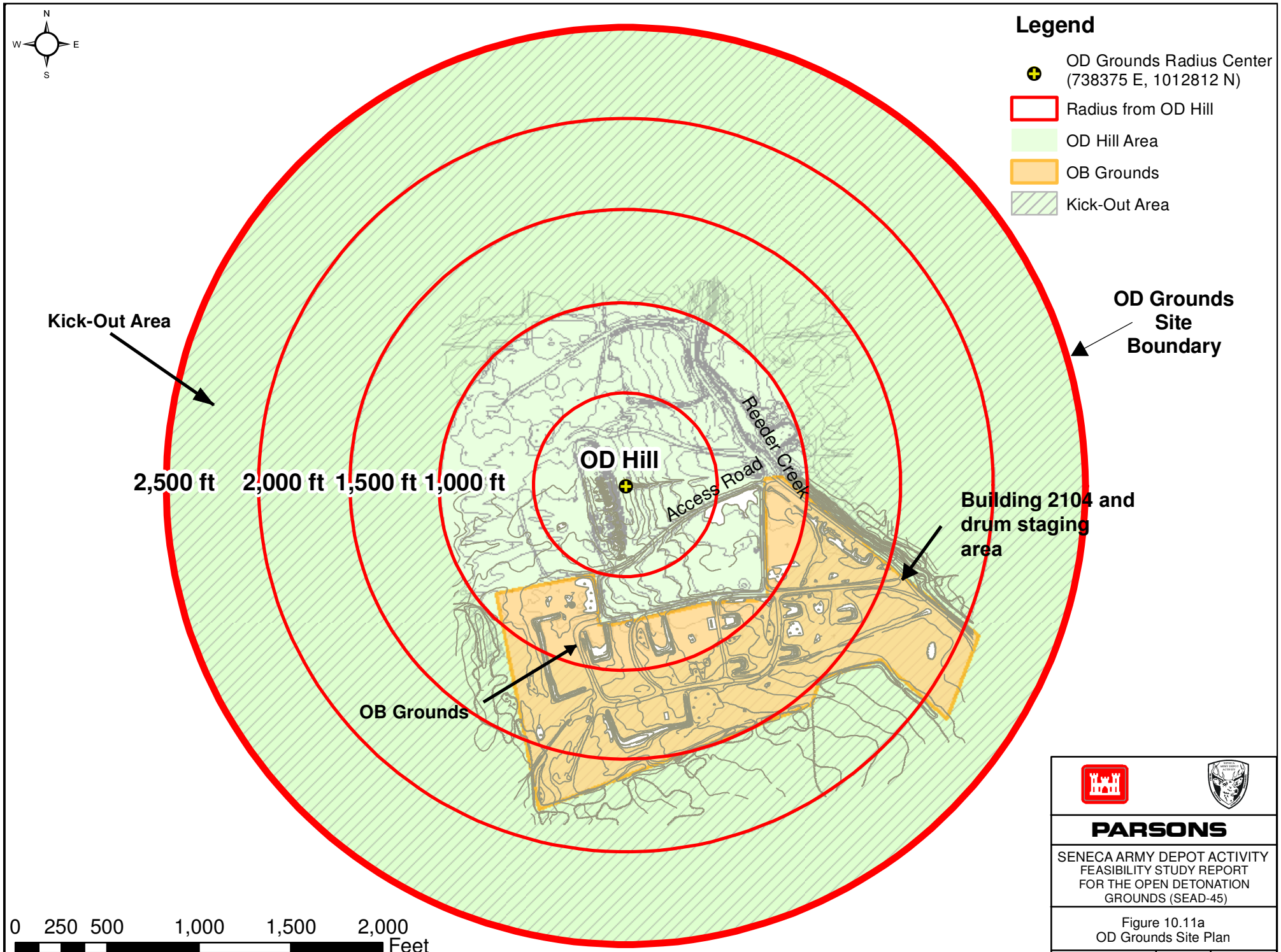
Table 10.3 - Overview of Preliminary Conceptual Site Model, SEAD-25, Seneca Army Depot Activity

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

Table 10.4 - Overview of Preliminary Conceptual Site Model, SEAD-26, Seneca Army Depot Activity

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

WORKSHEET #10 FIGURES



PARSONS

SENECA ARMY DEPOT ACTIVITY
FEASIBILITY STUDY REPORT
FOR THE OPEN DETONATION
GROUNDS (SEAD-45)

Figure 10.11a
OD Grounds Site Plan

July 2018	TIB	1:8,000

Path: P:\PIT\Projects\Huntsville Cont W912DY-08-D-0003\TO#13 - OD Grounds RI-FS\Documents\FS\DraftFinal FS\Figures_Revised\Figure 1-2.mxd

N
W E
S

MW3
MW2
MW45-1
MW45-2
MW45-3
MW4
MW45-4
MW1
MW5
MW23-4
MW23-5
MW23-6
MW23-1
MW23-2
MW23-3

622
626
620
624
628

GW Flow

OD Grounds
2500 FT Radius

0 125 250 500 750 1,000
Feet

Legend

- Long-Term Monitoring Well (2007-2012)
- Monitoring Well Sampled in 1995 ESI
- Drainage Pathways
- Reeder Creek
- OD Grounds Boundary
- OB Grounds Boundary
- Proposed Sampling Location

Note: OD Grounds groundwater contours are based on data collected from 4/1994 (Parsons, 1995).

Note: Proposed sample locations are circled. Existing wells will be sampled where possible. A maximum of 9 locations will be sampled. Dependent on the condition of the existing ESI wells (blue), up to four replacement points will be installed, as necessary. One sample will be collected from either MW4 or MW45-4.

SENECA ARMY DEPOT ACTIVITY

OPEN DETONATION GROUNDS (SEAD-45)

Figure 10.11b
Proposed Perchlorate Groundwater Sampling Locations at the OD Grounds

July 2018	1 in = 300 ft	TIB

Path: P:\PITProjects\Huntsville Cont W912DY-08-D-0003\TO#13 - OD Grounds RI-FS\Documents\Perchlorate Sampling\Work Plan\Draft Report\Figures\Figure 1 - GW Sampling Locations.mxd

N
W
E
S

S45-ODG-SS-01
S45-ODG-SS-04
S45-ODG-SS-05
S45-ODG-SS-09
S45-ODG-SS-08
S45-ODG-SS-03
S45-ODG-SS-07
S45-ODG-SS-06
S45-ODG-SS-02
S45-ODG-SS-10

Legend

- Proposed Soil Sample Location
- Drainage Pathways
- Reeder Creek
- OB Grounds Boundary

Note: Soil samples on the OD Hill will be adjusted in the field. Two samples will be located on top of the OD Hill, one sample will be located within a former demo pit, and one sample will be located on the side slope of the OD Hill.

Note: Two soil samples will be collected from each location. One sample from 0-6 inches bgs and a second sample between 6-12 inches bgs.

0 125 250 500 750 1,000 Feet

SENECA ARMY DEPOT ACTIVITY

OPEN DETONATION GROUNDS (SEAD-45)

Figure 10.11c
Proposed Perchlorate Soil Sampling Locations at the OD Grounds

May 2018	1 in = 200 ft	TIB

Path: P:\PIT\Projects\Huntsville Cont W912DY-08-D-0003\TO#13 - OD Grounds RI-FS\Documents\Perchlorate SamplingWork Plan\Figures\Figure 2 - Soil Sampling Locations.mxd

N
E
S
W

Non-Combustible Fill Landfill (NCFL)
(SEAD-8)
3.4 Acres

Ash Landfill
(SEAD-6)
2.2 Acres

MW-39
MW-43
MW-40
MW-60
PT-18A
MW-44A
MWT-25
1000
100
10
MWT-12R
MWT-26
MWT-27
MWT-28
PT-12A
MW-46
MWT-17R
MWT-22
MWT-29
PT-19
PT-21A
PT-20
PT-22
MW-48
MWT-23
MW-32
MW-33
PT-17
MWT-24
MW-27
MW-28
MWT-7,
-8, -9
MWT-7
MWT-4,
-5, -6
MW-29
MWT-1,
-2, -3
PT-16
MWT-10
PT-24
MW-56

0 150 300 600
Feet

Legend

- Double Biowall
- Pilot Biowall
- Single Biowall
- ZVI Wall
- TCE Isocontour (ppm) based on Jan 2000 data
- SEAD Locations
- Access Roads
- Paved Road
- Fenceline
- Railways
- SEDA Property Boundary
- Existing Well to Sample for 1,4-Dioxane
- Other Monitoring Wells

PARSONS

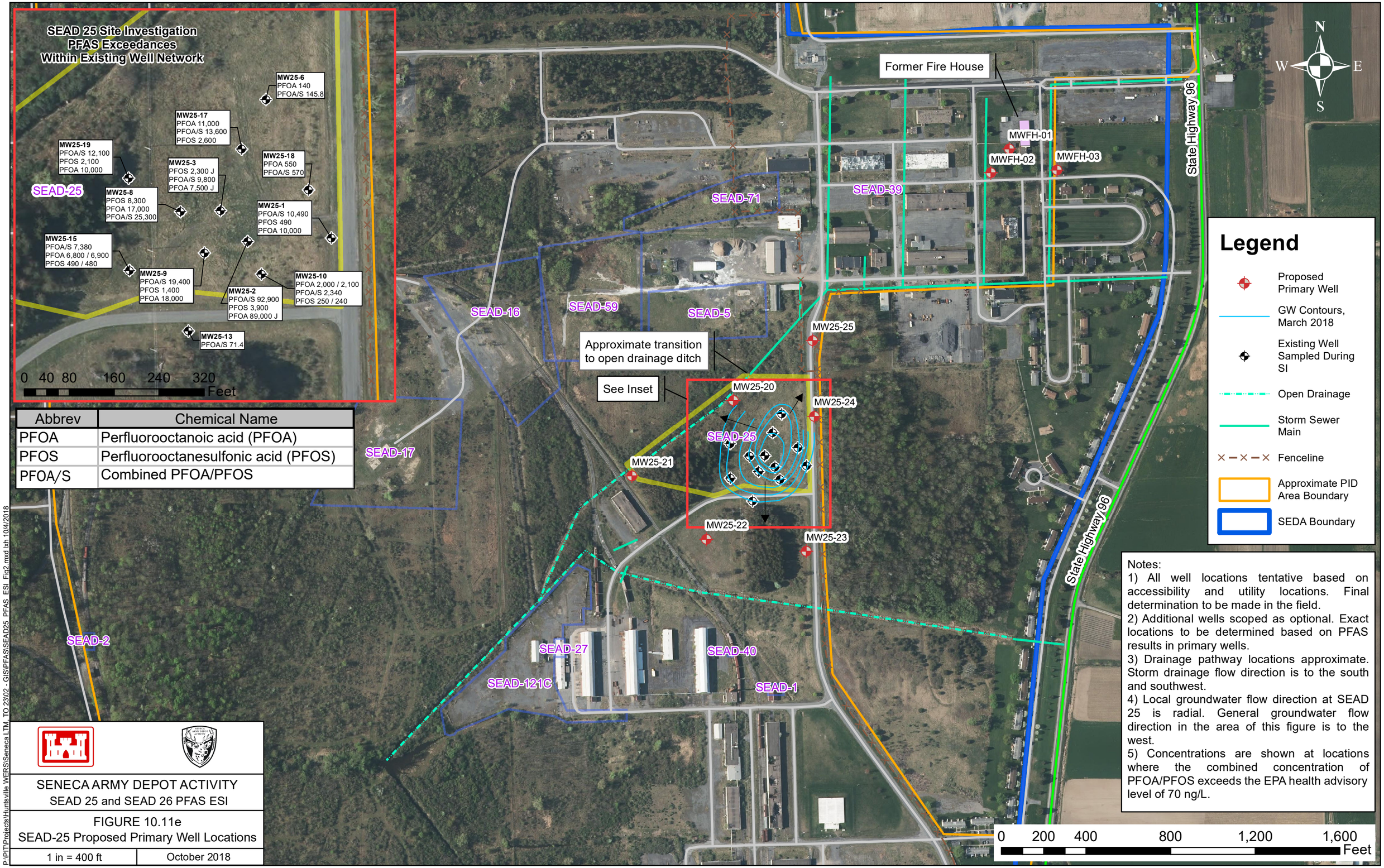
SENECA ARMY DEPOT ACTIVITY

Ash Landfill Groundwater Investigation

FIGURE 10.11d - Proposed Monitoring Well Network for 1,4-Dioxane Sampling

1 inch = 200 feet | August 2018

Path: P:\PIT\Projects\Huntsville WERS\Seneca LTM, TO 23102 - GIS\AshLandfill\1_4_Dioxane\Figure3-Sample Locs.mxd



Abbrev	Chemical Name
PFOA	Perfluorooctanoic acid (PFOA)
PFOS	Perfluorooctanesulfonic acid (PFOS)
PFOA/S	Combined PFOA/PFOS

Notes:
1) All well locations tentative based on accessibility and utility locations. Final determination to be made in the field.
2) Additional wells scoped as optional. Exact locations to be determined based on PFAS results in primary wells.
3) Drainage pathway locations approximate. Storm drainage flow direction is to the south and southwest.
4) Local groundwater flow direction at SEAD 25 is radial. General groundwater flow direction in the area of this figure is to the west.
5) Concentrations are shown at locations where the combined concentration of PFOA/PFOS exceeds the EPA health advisory level of 70 ng/L.

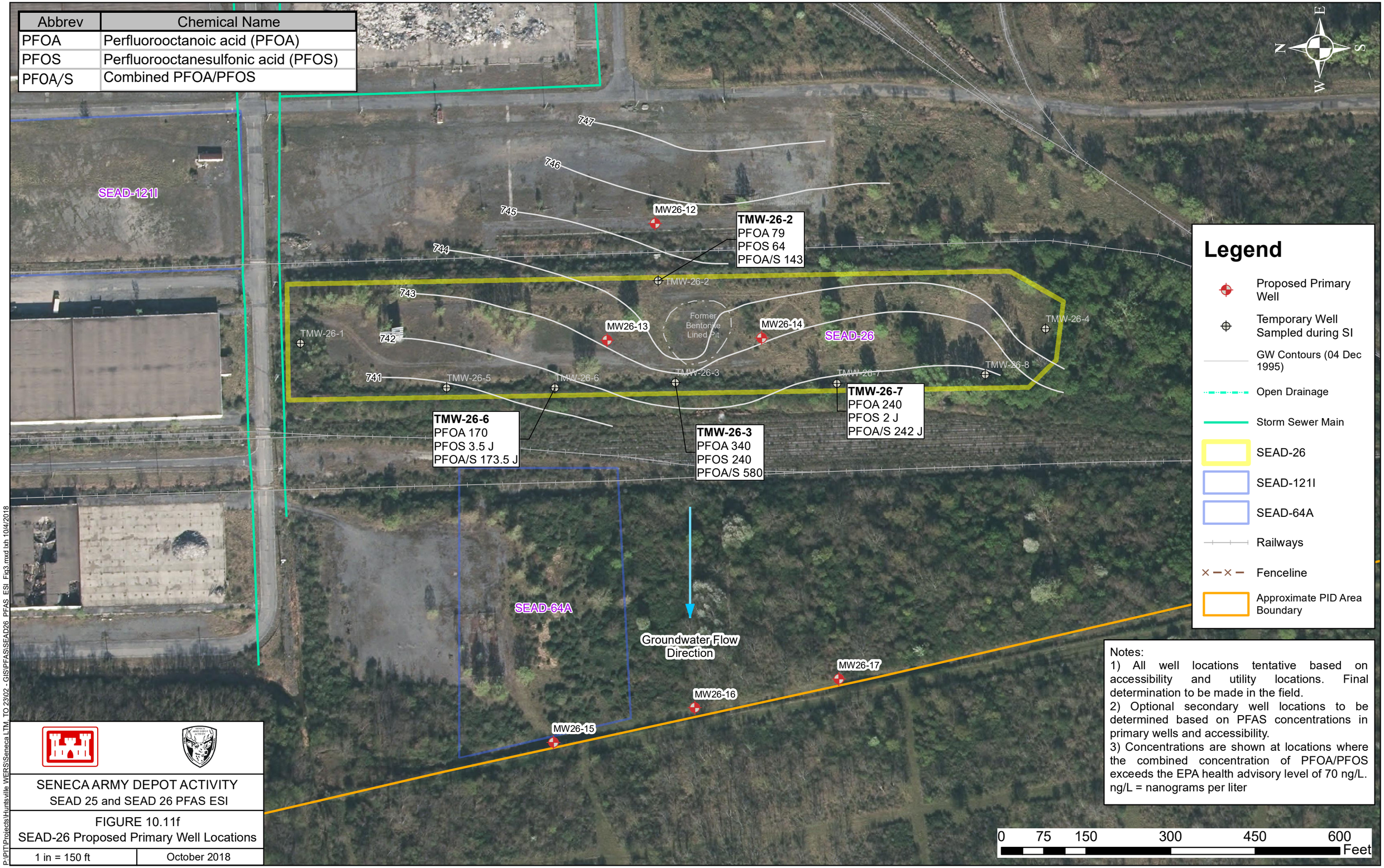
SENECA ARMY DEPOT ACTIVITY
SEAD 25 and SEAD 26 PFAS ESI

FIGURE 10.11e
SEAD-25 Proposed Primary Well Locations

1 in = 400 ft	October 2018

P:\PIT\Projects\Huntsville WERS\Seneca LTM_TO 23\02 - GIS\PFAS\SEAD25_PFAS_ESI_Fig2.mxd lxh 10/4/2018

Abbrev	Chemical Name
PFOA	Perfluorooctanoic acid (PFOA)
PFOS	Perfluorooctanesulfonic acid (PFOS)
PFOA/S	Combined PFOA/PFOS



Legend

- Proposed Primary Well
- Temporary Well Sampled during SI
- GW Contours (04 Dec 1995)
- Open Drainage
- Storm Sewer Main
- SEAD-26
- SEAD-121I
- SEAD-64A
- Railways
- Fenceline
- Approximate PID Area Boundary

Notes:
1) All well locations tentative based on accessibility and utility locations. Final determination to be made in the field.
2) Optional secondary well locations to be determined based on PFAS concentrations in primary wells and accessibility.
3) Concentrations are shown at locations where the combined concentration of PFOA/PFOS exceeds the EPA health advisory level of 70 ng/L. ng/L = nanograms per liter

0 75 150 300 450 600 Feet

SENECA ARMY DEPOT ACTIVITY
SEAD 25 and SEAD 26 PFAS ESI

FIGURE 10.11f
SEAD-26 Proposed Primary Well Locations

1 in = 150 ft	October 2018

P:\PIT\Projects\Huntsville WERS\Seneca LTM_TO 23J02 - GIS\PFAS\SEAD26_PFAS_ESI_Fig3.mxd lxh 10/4/2018

Worksheet #11: Data Quality Objectives

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

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

The purpose of the perchlorate soil and groundwater investigation is to determine the presence or absence of perchlorate in soil and groundwater as a result of ordnance detonation activities. Soil sampling locations were selected to be near the OD Hill (source) and in nearby drainage ditches. The purpose of the 1,4-dioxane groundwater sampling is to determine the presence or absence of this compound at the Ash Landfill. Additional groundwater sampling for PFAS at SEAD 25 and SEAD 26 will further delineate the area of contamination. Specific DQOs for each site are outlined in **Table 11.1**. These DQOs follow the USEPA's seven-step, iterative process for DQO development. Based on the overall goal, the general project DQOs are to obtain data to sufficiently characterize the soil and groundwater concentrations at each site.

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

Table 11.1 - Data Quality Objectives and Technical Approach Summary for Emerging Contaminant Sampling at SEDA

SITE	STATE THE PROBLEM	IDENTIFY THE GOAL OF THE STUDY	IDENTIFY INFORMATION INPUTS	DEFINE THE BOUNDARIES OF THE STUDY	DEVELOP THE ANALYTIC APPROACH	SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA (SEE WORKSHEET #17)	DEVELOP THE DETAILED PLAN FOR OBTAINING DATA (SEE WORKSHEET #17)
OD Grounds	• Perchlorate is an emerging contaminant and may have a potential impact on human health and the environment	• Determine the presence or absence of perchlorate in soil and groundwater as a result of munitions disposal activities.	• Analytical groundwater data (perchlorate) • Analytical soil data (perchlorate)	• The investigation will be in the area surrounding OD Hill. • This is a site investigation to determine if perchlorate is present. If perchlorate concentrations exceed the acceptance criteria, further action may be proposed.	• Review groundwater concentrations for perchlorate (Worksheet #17). Evaluation of potential contamination and recommendations for future actions. • Review soil concentrations for perchlorate. Evaluation of potential contamination and recommendations for future actions.	• EPA RSLs (See Table 15.2 and 15.3)	• Collect groundwater samples via low-flow techniques in existing monitoring wells. • Collect soil samples utilizing a hand auger at 10 locations.
Ash Landfill	• 1,4-dioxane is an emerging contaminant and may have a potential impact on human health and the environment	• Determine the presence or absence of 1,4-dioxane in groundwater based on the presence of chlorinated VOCs at the site.	• Analytical groundwater data (1,4-dioxane)	• The investigation will be at existing monitoring wells. The wells sampled will be detailed in a letter-style workplan issued prior to the sampling event. • This is a site investigation to determine if 1,4-dioxane is present. If 1,4-dioxane concentrations exceed the acceptance criteria, further action may be proposed.	• Review groundwater concentrations for1,4-dioxane (Worksheet #17). Evaluation of potential contamination and recommendations for future actions.	• EPA RSLs (See Table 15.1)	• Collect groundwater samples using low-flow techniques at existing monitoring wells.
SEAD-25	• PFAS are an emerging contaminant and have a potential impact on human health and the environment	• Determine the presence or absence of PFAS in groundwater as a result of firefighting training activities	• Analytical groundwater data (PFAS)	• The investigation will be around the perimeter of the former fire training area and the former fire house. • This is a site investigation to determine if PFAS are present. If PFAS concentrations exceed the acceptance criteria, further action may be proposed.	• Review groundwater concentrations for PFAS COCs (Worksheet #17). Evaluation of potential contamination and recommendations for future actions.	• EPA temporary provisional health advisory level (See Table 15.4)	• Collect groundwater samples from new, 2-inch monitoring wells using low-flow techniques
SEAD-26	• PFAS are an emerging contaminant and have a potential impact on human health and the environment	• Determine the presence or absence of PFAS in groundwater as a result of firefighting training activities	• Analytical groundwater data (PFAS)	• The investigation will be around the perimeter of the former fire training area. • This is a site investigation to determine if PFAS are present. If PFAS concentrations exceed the acceptance criteria, further action may be proposed.	• Review groundwater concentrations for PFAS COCs (Worksheet #17). Evaluation of potential contamination and recommendations for future actions.	• EPA temporary provisional health advisory level (See Table 15.4)	• Collect groundwater samples from new, 2-inch monitoring wells using low-flow techniques

Worksheet #12: Measurement Performance Criteria

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

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

12.1 MEASUREMENT PERFORMANCE CRITERIA FOR SVOCS IN GROUNDWATER

Laboratory: Katahdin
Matrix: Groundwater
Analytical Group or Method: SVOC/ SW8270D SIM
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	Relative Percent Difference (RPD) ≤ 30% when SVOCs are detected in both samples with concentrations are ≥ sample specific Limit of Quantitation (LOQ). If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 40%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Within DoD Quality Systems Manual (QSM) Version 5.1 Appendix B, Table 28 limits or laboratory limits if not listed in Table 28 of Appendix B.
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Within DoD Quality Systems Manual (QSM) Version 5.1 Appendix B, Table 28 limits or laboratory limits if not listed in Table 28 of Appendix B.
Overall accuracy/bias (contamination)	Method Blanks/Equipment Blanks	No target analyte concentrations ≥ 1/2 LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater
Sensitivity	LOQ verification sample (spiked at LOQ)	Recovery within ±25% of LOQ
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

12.2 MEASUREMENT PERFORMANCE CRITERIA FOR PERCHLORATE IN GROUNDWATER AND SOIL

Laboratory: TestAmerica - Denver
Matrix: Groundwater and Soil
Analytical Group or Method: Perchlorate by SW6860
Concentration Level Low

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	RPD ≤ 30% for waters and RPD ≤ 50% for soils when Perchlorate is detected in both samples with concentrations are ≥ sample specific LOQ). If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤15%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Within DoD QSM Version 5.1 Appendix B Table 7 (soil) and Table 8 (water) limits
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Within DoD QSM Version 5.1 Appendix B Table 7 (soil) and Table 8 (water) limits
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ 1/2 LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater
Sensitivity	LOQ verification sample (spiked at LOQ)	Recovery within ±25% of LOQ
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

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

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

DATA QUALITY INDICATORS	QC SAMPLE OR MEASUREMENT PERFORMANCE ACTIVITY	MEASUREMENT PERFORMANCE CRITERIA
Overall Precision	Field Duplicates	RPD ≤ 30% when the analyte is detected in both samples ≥ sample-specific LOQ. If one result is > LOQ and the other ND, "J" flag the detected result and "UJ" the ND result. If one result is >LOQ and the other result is <LOQ, "J" flag will be applied to the result >LOQ
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates, Matrix Spike Duplicates	RPD ≤ 30%
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	See Worksheet #28, Table 28.3b
Analytical Accuracy/Bias (matrix interference)	Matrix Spike	See Worksheet #28, Table 28.3b
Overall accuracy/bias (contamination)	Equipment Blanks, Trip Blanks and Field Blanks	No target analyte concentrations ≥ 1/2 LOQ
Sensitivity	LOQ verification sample (spiked at LOQ)	Performed quarterly per the requirements included in the DoD QSM, version 5.1
Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check

Worksheets #14 & 16: Project Tasks and Schedule

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

The additional activities to be conducted at Seneca Army Depot Activity to achieve the project DQOs (**Worksheet #11**) comprise of one primary component: to obtain analytical data to monitor the presence or absence of perchlorate, 1,4-dioxane, and PFAS compounds at the OD Grounds, Ash Landfill and SEADs 25 and 26. Multiple elements, or "DFWs," are required to achieve the project goals. This subchapter provides a summary of these DFWs and the associated component tasks. A detailed discussion of the primary project component at each site and the related DFWs is included on **Worksheet #17**, and the specific field procedures to be used for the activities described in this summary are included in the various SOPs appended to this UFP-QAPP. The project schedules will be provided in site specific planning documents.

DEFINABLE FEATURE OF WORK (ACTIVITY)	ASSOCIATED TASKS	RELATED SOPS
Mobilization	• Preparation (review plans, make travel arrangements, etc.) • Mobilize equipment and vehicles to the site • Set up site communications • Conduct site-specific training and briefing for required field personnel	--
Site Preparation	• Set up and calibrate sampling equipment • Prepare sample bottles and labels	--
Sampling and Analysis	• Collect and analyze groundwater samples • Conduct QC evaluation of analytical data for validation • Document data validation and sample results	• Parsons SOPs (Worksheet #21): • Analytical SOPs (Worksheet #23) • Parsons Modified PFAS SOPs (work plan)
Demobilization	• Upon completion of field activities all personnel, equipment and materials will be removed from the site	--
Reports	• OD Grounds: The results of the perchlorate investigation will be reported in the OD Grounds Feasibility Report. • Ash Landfill: Site inspection report presenting a summary of completed field activities, summary of data, including presentation on tables and figures, and evaluation of contamination and recommendations for future actions. • SEAD 25/26: Expanded site inspection report to provide a summary of completed field activities, summary of data, including presentation on tables and figures, and evaluation of contamination and recommendations for future actions.	

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

(EPA UFP-QAPP Guidance Manual, Section 2.8.1)

This worksheet provides the parameters to be analyzed and their associated limits of quantitation (LOQ), limits of detection (LOD), and detection limits (DL) in order to satisfy the overall DQOs. The PALs, as referenced in the DQOs on **Worksheet #11**, are also included. The Project Action Limits (PALs) for this project were selected based on the lowest enforceable standard between the New York State Class GA (NYS Class GA) Ambient Water Quality Standards (NYSDEC, 2018) and the USEPA Regional Screening Levels (RSL) (EPA, 2018).

Table 15.1 - Project Action Limits and Katahdin Reference Limits for SVOCs in Groundwater (Method SW-846 8270D SIM)

ANALYTE	PROJECT ACTION LIMIT (µG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (3) (µG/L)	LOD (µG/L)	DL (µG/L)
1,4-Dioxane	0.46	RSL	0.25	0.18	0.085
	0.35	EPA HA (2)			

(1) No NYSDEC value is available. PAL was selected from EPA Regional Screening Level (RSL) Summary Table (TR=1E-06, HQ=0.1) May 2018. https://semspub.epa.gov/work/HQ/197235.pdf

(2) The EPA Health Advisory level was provided for informational purposes. EPA risk assessments indicate that the drinking water concentration representing a 1 x 10-6 cancer risk level for 1,4-dioxane is 0.35 µg/L (EPA IRIS 2013). Technical Fact Sheet – 1,4-Dioxane, November 2017. https://www.epa.gov/sites/production/files/2014-03/documents/ffrro_factsheet_contaminant_14-dioxane_january2014_final.pdf

Table 15.2 - Project Action Limits and TestAmerica Denver Reference Limits for Perchlorate in Groundwater (Method SW-846 6860)

ANALYTE	PROJECT ACTION LIMIT (µG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (µG/L)	LOD (µG/L)	DL (µG/L)
Perchlorate	1.4	RSL	0.0500	0.0100	0.00400

(1) No NYSDEC value is available. PAL was selected from EPA Regional Screening Level (RSL) Summary Table (TR=1E-06, HQ=0.1) May 2018. https://semspub.epa.gov/work/HQ/197235.pdf

Table 15.3 - Project Action Limits and TestAmerica Denver Reference Limits for Perchlorate in Soil (Method SW-846 6860)

ANALYTE	PROJECT ACTION LIMIT (µG/KG) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (µG/KG)	LOD (µG/KG)	DL (µG/KG)
Perchlorate	5,500	RSL	0.500	0.100	0.0400

(1) No NYSDEC value is available. PAL was selected from EPA Regional Screening Level (RSL) Summary Table (TR=1E-06, HQ=0.1) May 2018. https://semspub.epa.gov/work/HQ/197235.pdf

Table 15.4 - Project Action Limits and TestAmerica- W. Sacramento Reference Limits for PFAS (EPA Method 537 mod)

ANALYTE	PROJECT ACTION LIMIT (nG/L) (1)	PAL REFERENCE	ACHIEVABLE LABORATORY LIMITS		
			LOQ (nG/L)	LOD (nG/L)	DL (nG/L)
Perfluorohexanoic acid (PFHxA)	NA	NA	2.00	1.00	0.470
Perfluoroheptanoic acid (PFHpA)	NA	NA	2.00	1.50	0.610
Perfluorooctanoic acid (PFOA)	70	EPA Health Advisory Limit (2)	2.00	1.50	0.540
Perfluorononanoic acid (PFNA)	NA	NA	2.00	1.50	0.520
Perfluorodecanoic acid (PFDA)	NA	NA	2.00	1.00	0.480
Perfluoroundecanoic acid (PFUnA)	NA	NA	2.00	1.50	0.720
Perfluorododecanoic acid (PFDoA)	NA	NA	2.00	1.50	0.520
Perfluorotridecanoic Acid (PFTriA)	NA	NA	4.00	3.00	0.760
Perfluorotetradecanoic acid (PFTeA)	NA	NA	4.00	3.00	0.830
Perfluorobutanesulfonic acid (PFBS)	NA	NA	2.00	1.00	0.460
Perfluorohexanesulfonic acid (PFHxS)	NA	NA	2.00	1.00	0.380
Perfluorooctanesulfonic acid (PFOS)	70	EPA Health Advisory Limit (2)	4.00	3.00	1.10
N-ethyl perfluorooctane sulfonamidoacetic acid (NEtFOSAA)	NA	NA	20.0	10.0	2.80
N-methyl perfluorooctane sulfonamidoacetic acid (NMeFOSAA)	NA	NA	20.0	10.0	3.00
Perfluorobutanoic acid (PFBA)			2.00	1.50	0.590
Perfluoropentanoic acid (PFPeA)			2.00	1.00	0.430
Perfluoroheptanesulfonic Acid (PFHpS)			2.00	1.00	0.370
Perfluorodecanesulfonic acid (PFDS)			2.00	1.50	0.560
Perfluorooctane Sulfonamide (PFOSA)			4.00	3.00	1.30
6:2 FTS			40.0	20.0	7.00
8:2 FTS			20.0	10.0	3.00

(1) NA – No NYSDEC Class GA or EPA MCL available.

(2) EPA Health Advisory Limits are drinking water limits (EPA, 2016b). The PAL applies to the concentration of a single compound (e.g, PFOS) or combined concentration (PFOA + PFOS).

Worksheet #17: Sampling Design and Rationale

(EPA UFP-QAPP Guidance Manual, Section 3.1.1)

17.1 INTRODUCTION

Sampling for perchlorate will be conducted at the Seneca Open Detonation Grounds (OD Grounds) and 1,4-dioxane at the Ash Landfill. Sampling for PFAS will be conducted at SEAD 25 and SEAD 26. The boundaries of the each site are shown on **Figures 10.11a** through **10.11f**, respectively. The general technical approach is based on the CSM for each Site, which is described on **Worksheet #10**. Full details of the sampling design will be provided in individual letter style work plans specific to perchlorate sampling at OD Grounds, 1,4-dioxane sampling at the Ash Landfill, and PFAS sampling at SEAD 25 and SEAD 26.

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

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

17.2 DEFINEABLE FEATURES OF WORK

17.2.1 MOBILIZATION

Preparations for mobilization will commence upon approval of this UFP-QAPP and Addendum. Perchlorate and PFAS sampling will be conducted under separate mobilization. Mobilization for 1,4-dioxane sampling may be combined with the LTM sampling at Ash Landfill. Upon receipt of document approval, the field team will be notified, travel and lodging arrangements will be made, and the requisite copies of applicable documents will be assembled. The field management team will have already reviewed the available documentation relating to the site and this UFP-QAPP. Based on historic data and findings, the field teams will be mobilized to Seneca around the following timeframes as presented in **Table 17.1** below to ensure optimal sampling conditions.

Table 17.1 – Mobilization Schedule

SITE	SAMPLE COLLECTION TIMEFRAME (1)
Ash Landfill	June, December
SEAD 25 & SEAD 26	November
OD Grounds	June

(1) Timeframes may shift if deemed appropriate or necessary

Equipment and materials will either be shipped to the site via commercial carrier, transported to the site by the field team, or obtained locally, as appropriate. Equipment may include, but is not limited to, sampling supplies, sample containers, documents, first aid kits, fire extinguishers, digital cameras, etc. Site vehicles will be rented and, in most cases, will be four-wheel drive vehicles that will accommodate all site personnel and equipment. Drilling equipment will be brought to the site by a subcontractor.

The primary means of onsite communication will be achieved using cellular telephones. If separated from one another, each member of the field sampling team will have an operational cell phone available at all times for emergency use. Additional information can be found in the Accident Prevention Plan (APP) / Site Safety and Health Plan (SSHP) (Parsons, 2017).

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

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

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

17.2.2 SITE PREPARATION

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

17.2.3 SAMPLING AND ANALYSIS

Additional sites will be sampled for the presence or absence of perchlorate and 1,4-dioxane. In addition to the sampling and analysis descriptions provided below for each site, the specific details are addressed in greater detail on **Worksheet #18** and in Parsons SOP ENV-02 and PFAS SOPs (**Appendix C, PFAS Work Plan**), and the analytical procedures are summarized on **Worksheets #19** and **#30** and **Worksheet #23**. Example field sampling forms are located in the Final UFP-QAPP (**Appendix B)**.

OD Grounds

No long-term monitoring has been conducted at the OD Grounds and perchlorate has not been sampled at this site. A total of nine monitoring wells and 20 soil samples are proposed as show on **Figures 10.11a+b**. Rationale for the number and locations of samples can be found in Section 3.1 and 3.3 of the Final Work Plan (Parsons, 2018c).

Sampling will be conducted at existing wells, that are in good condition as determined by a well condition survey conducted prior to sampling (Section 3.1 Final Work Plan, **Figure 10.11b**). In the event that the existing wells are no longer in good condition, new monitoring wells will be installed, as needed. The number of new monitoring wells installed will be based on the results of the well condition survey which is discussed in Section 3.1. Sampling will be conducted using low flow sampling methods. Groundwater samples will be collected from new monitoring wells installed using direct push techniques or from existing monitoring wells depending on their condition.

The wells will be purged and samples will be collected in accordance with SOP ENV-02 (excluding 5.3.3 and 5.3.4) (**Worksheet #21**). The groundwater samples will be analyzed for perchlorate using analytical method EPA SW846 Method 6860 (**Tables 15.2 and 15.3**). A comprehensive list showing analyses to be performed at each well is included on **Worksheet #18**.

Soil samples will be collected from two depths at ten locations (**Figure 10.11c**). The sample locations were selected to be biased to locations most likely to be impacted given the site history which included open detonation of various munitions with activity centered on the OD Hill. The highest concentration of munitions (which represent a potential source of contamination) remains within the shallow subsurface with the greatest quantities of debris just below the surface and decreasing amounts with increased depth. Therefore, samples were targeted at the potential source area (surface soil samples collected 0-6 inches below ground surface [bgs]) and just below this depth within the vadose zone above the seasonal water table (subsurface samples collected 18-24 inches bgs).

Surface and subsurface soil samples will be discrete and sampled in accordance with SOP ENV-01 (Sections 5.3.1, 5.3.2, 5.3.6, 5.4.1, 5.4.2, 5.4.3, 5.5) and **Worksheet #21**. Well IDs, sample locations and analyses are detailed in **Worksheet #18**.

Soil and groundwater concentrations will be reviewed for perchlorate COCs (**Worksheet #17**) and will be compared with EPA RSLs (**Worksheet #15**). A Site Investigation report will be written and will include evaluation of potential contamination and recommendations for future actions.

Ash Landfill

Currently, long-term monitoring at the Ash Landfill is focused on chlorinated solvents. By request from NYSDEC, the emerging contaminant 1,4-dioxane will be sampled in eight of the existing monitoring wells (MW-43, PT-18A, MW44A, MWT-25, MW-27, MWT-24, PT-20, and MW-56) at the Ash Landfill (Ref., Section 3.1 of the Work Plan and **Figure 10.11d**). The sampling program for 1,4-dioxane is detailed in a letter style work plan (Parsons, 2018d).

Groundwater concentrations will be reviewed for 1,4-dioxane (**Worksheet #17**) and will be compared with EPA RSLs (**Worksheet #15**) and if 1,4-dioxane concentrations exceed the performance criteria, further action will be discussed with the project team. A Site Investigation report will be written and will include evaluation of potential contamination and recommendations for future actions.

The wells will be purged and samples will be collected in accordance with SOP ENV-02 (excluding 5.3.3 and 5.3.4) (**Worksheet #21**). The groundwater samples will be analyzed for 1,4-dioxane using analytical method EPA SW846 Method 8270D-SIM (**Table 15.1**). A comprehensive list showing analyses to be performed at each well is included on **Worksheet #18**.

SEAD 25 and 26

Based on the results of the 2017 PFAS SI at SEADs 25, 26 and 122, the NYSDEC requested further investigation at SEAD 25 and SEAD 26. A letter style work plan details the specifics of the PFAS expanded site investigation (ESI) (Parsons, 2018e). The key elements of the PFAS ESI included the installation of new, 2-inch monitoring wells (Ref., Work Plan Section 3.1.1) and low flow groundwater sampling using modified drilling and groundwater sampling techniques (Ref., Work Plan Section 3.0) which are appropriate for PFAS investigations (see Work Plan PFAS specific SOPs) (**Figures 10.11e and 10.11f**). The groundwater samples will be analyzed for PFAS using Method 537M based on DoD QSM 5.1 (**Table 15.4**). A comprehensive list showing analyses to be performed at each well is included on **Worksheet #18**.

17.2.4 LUC INSPECTIONS

OD Grounds

There are currently no LUCs on the OD Grounds property.

SEAD 25, SEAD 26, Ash Landfill

LUCs per the Final UFP-QAPP, Section 17.2.4

17.2.5 DEMOBILIZATION

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

Worksheet #18: Sampling Locations and Methods

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

The sample rationale for the perchlorate, 1,4-dioxane and PFAS sampling at the OD Grounds, Ash Landfill and SEADs 25 and 26, respectively, is summarized in **Tables 18.1** through **18.5**. Perchlorate sampling locations at OD Grounds and 1,4-dioxane sample locations at Ash Landfill are shown in **Figures 10.11b , c and d, respectively.** PFAS sampling locations at SEAD 25 and SEAD 26 are shown in **Figures 10.11e + f**. Sample locations and final sample IDs for 1,4-dioxane at Ash Landfill and PFAS at SEADs 25 and 26 were provided in a letter style work plan. Sample ID nomenclature is explained in **Worksheet #26**. Sample locations and IDs are provided in **Tables 18.1 through 18.4**.

Table 18.1 – Groundwater Sampling Locations and Methods for Perchlorate at OD Grounds

LOCATION ID	SAMPLE ID(1)	MATRIX	SCREEN DEPTH (FT BGS)	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
MW1	45FS20001	GW	7-12	Sample	Perchlorate	ENV-02	
MW2	45FS20002	GW	1-6	Sample	Perchlorate	ENV-02	
MW3	45FS20003	GW	4.5-9.5	Sample	Perchlorate	ENV-02	
MW45-1	45FS20004	GW	3.25-5.25	Sample	Perchlorate	ENV-02	
MW45-2	45FS20005	GW	4.33-9.33	Sample	Perchlorate	ENV-02	
MW45-2	45FS20005MS	GW	4.33-9.33	MS/MSD	Perchlorate	ENV-02	
MW45-2	45FS20005MSD	GW	4.33-9.33	MS/MSD	Perchlorate	ENV-02	
MW45-2	45FS20006	GW	4.33-9.33	Field Duplicate	Perchlorate	ENV-02	
MW45-3	45FS20007	GW	5.58-10.58	Sample	Perchlorate	ENV-02	
MW45-4	45FS20008	GW	4.25-6.25	Sample	Perchlorate	ENV-02	
MW23-3	45FS20009	GW	6.9-11.9	Sample	Perchlorate	ENV-02	
MW23-4	45FS20010	GW	9-14	Sample	Perchlorate	ENV-02	

Key: GW = groundwater;

(1) One MS/MSD and one field duplicate will be collected. The locations of the MS/MSD and field duplicate will be determined in the field based on site conditions. One of the existing sample IDs in the table above will be appended with MS and MSD (e.g., 45FS20005MS and 45FS20005MSD). The field duplicates will be collected at the same location as the MS/MSD and the sample ID will be one larger than the last ID shown in the table (e.g., 45FS20006).

Table 18.2 – Soil Sampling Locations and Methods for Perchlorate at OD Grounds

GEOGRAPHIC LOCATION	LOCATION ID	MATRIX	SAMPLE ID(1)	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	ALTERNATIVE SAMPLE ID
Drainage	S45-ODG-SS-01	SS	45-FS-SS-10001-0-0.5	SA	Perchlorate	ENV-02	
Drainage	S45-ODG-SB-01	SB	45-FS-SB-10001-1.5-2.0	SA	Perchlorate	ENV-02	
Drainage	S45-ODG-SS-02	SS	45-FS-SS-10002-0-0.5	SA	Perchlorate	ENV-02	
Drainage	S45-ODG-SB-02	SB	45-FS-SB-10002-1.5-2.0	SA	Perchlorate	ENV-02	
Kickout	S45-ODG-SS-03	SS	45-FS-SS-10003-0-0.5	SA	Perchlorate	ENV-02	45-FS-SS-WellID-10003-0-0.5
Kickout	S45-ODG-SB-03	SB	45-FS-SB-10003-1.5-2.0	SA	Perchlorate	ENV-02	45-FS-SB-WellID-10003-1.5-2.0
Kickout	S45-ODG-SS-04	SS	45-FS-SS-10004-0-0.5	SA	Perchlorate	ENV-02	45-FS-SS-WellID-10003-0-0.5
Kickout	S45-ODG-SB-04	SB	45-FS-SB-10004-1.5-2.0	SA	Perchlorate	ENV-02	45-FS-SB-WellID-10003-1.5-2.0
OD Hill	S45-ODG-SS-05	SS	45-FS-SS-10005-0-0.5	SA	Perchlorate	ENV-02	
OD Hill	S45-ODG-SB-05	SB	45-FS-SB-10005-1.5-2.0	SA	Perchlorate	ENV-02	
OD Hill	S45-ODG-SS-06	SS	45-FS-SS-10006-0-0.5	SA	Perchlorate	ENV-02	
OD Hill	S45-ODG-SB-06	SB	45-FS-SB-10006-1.5-2.0	SA	Perchlorate	ENV-02	
OD Hill	S45-ODG-SS-07	SS	45-FS-SS-10007-0-0.5	SA	Perchlorate	ENV-02	
OD Hill	S45-ODG-SB-07	SB	45-FS-SB-10007-1.5-2.0	SA	Perchlorate	ENV-02	
OD Hill	S45-ODG-SS-08	SS	45-FS-SS-10008-0-0.5	SA	Perchlorate	ENV-02	
OD Hill	S45-ODG-SB-08	SB	45-FS-SB-10008-1.5-2.0	SA	Perchlorate	ENV-02	
Kickout	S45-ODG-SS-09	SS	45-FS-SS-10009-0-0.5	SA	Perchlorate	ENV-02	45-FS-SS-WellID-10003-0-0.5
Kickout	S45-ODG-SB-09	SB	45-FS-SB-10009-1.5-2.0	SA	Perchlorate	ENV-02	45-FS-SB-WellID-10003-1.5-2.0
Kickout	S45-ODG-SS-10	SS	45-FS-SS-10010-0-0.5	SA	Perchlorate	ENV-02	45-FS-SS-WellID-10010-0-0.5
Kickout	S45-ODG-SB-10	SB	45-FS-SB-10010-1.5-2.0	SA	Perchlorate	ENV-02	45-FS-SB-WellID-10010-1.5-2.0
TBD	TBD	SB	45-FS-SB-10011-1.5-2.0	DU	Perchlorate	ENV-02	45-FS-SB-WellID-10011-1.5-2.0
TBD	TBD	SB	45-FS-SB-10011-1.5-2.0	DU	Perchlorate	ENV-02	45-FS-SB-WellID-10011-1.5-2.0
N/A	N/A	AQ	45-FS-00001	EB	Perchlorate	ENV-02	

Key: SS = Surface soil; SB = Subsurface Soil; SA = Sample; DU = Duplicate; MS/MSD = Matrix Spike / Duplicate; EB = Equipment Blank

1) One MS/MSD and two field duplicates will be collected. The locations of the MS/MSD and field duplicate will be determined in the field based on site conditions. One of the existing sample IDs in the table above will be appended with MS and MSD (e.g., 45-FS-SB-MW45-1R-10004-1.5-2.0MS and 45-FS-SB-MW45-1R-10004-1.5-2.0MSD). The field duplicates will be collected at the same location as the MS/MSD and the sample ID will be one larger than the last ID shown in the table (e.g., 45-FS-SB-10011-1.5-2.0).
2) Red depth intervals (feet) will be changed in the field and will correspond to the interval the soil sample was taken. Hand augured locations will be taken as close as possible to the vadose zone / water table interface.
3) If soil borings are conducted, a sample may be collected from the boring. The sampling locations noted with Alternative IDs will be appended with associated soil boring (e.g, 45-FS-SS-MW45-1R-10004-0-0.5 and 45-FS-SB-MW45-1R-10004-1.5-2.0).

Table 18.3 – Groundwater Sampling Locations and Methods for 1,4-Dioxane at Ash Landfill

LOCATION ID[2]	SAMPLE ID[1]	MATRIX	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
MW-43	ALMI20001	GW	Sample	1,4-Dioxane	ENV-02, 5.3.2	
PT-18A	ALMI20002	GW	Sample	1,4-Dioxane	ENV-02, 5.3.2	LTM Well
MW-44A	ALMI20003	GW	Sample	1,4-Dioxane	ENV-02, 5.3.2	
MWT-25	ALMI20004	GW	Sample	1,4-Dioxane	ENV-02, 5.3.2	LTM Well
MW-27	ALMI20005	GW	Sample	1,4-Dioxane	ENV-02, 5.3.2	
MWT-24	ALMI20006	GW	Sample	1,4-Dioxane	ENV-02, 5.3.2	LTM Well
PT-20	ALMI20007	GW	Sample	1,4-Dioxane	ENV-02, 5.3.2	LTM Well
MW-56	ALMI20008	GW	Sample	1,4-Dioxane	ENV-02, 5.3.2	Off-site Well
TBD	ALMI2000#MS	GW	MS/MSD	1,4-Dioxane	ENV-02, 5.3.2	
TBD	ALMI2000#MSD	GW	MS/MSD	1,4-Dioxane	ENV-02, 5.3.2	
TBD	ALMI20009	GW	Duplicate	1,4-Dioxane	ENV-02, 5.3.2	
N/A	ALMI00001	Aqueous	Rinse Blank	1,4-Dioxane	ENV-02, 5.3.2	

Key: GW = groundwater;

(1) MS/MSD and Field Duplicate locations will be determined in the field, as groundwater conditions allow.

(2) Approximate number of samples shown. Locations are to be determined but will be from existing wells at the Ash Landfill and detailed in a letter style work plan issued prior to sampling.

(3) Reference 1,4-dioxane Groundwater Investigation Work Plan (Parsons, 2018d)

Table 18.4 – Sampling Locations and Methods for PFAS at SEAD-25 and SEAD 26

LOCATION ID	SAMPLE ID(1)	MATRIX	SCREEN DEPTH (FT BGS)6	TYPE	ANALYTE / ANALYTICAL GROUP	SAMPLING SOP	COMMENTS
SEAD 25 Sample Locations and Nomenclature							
MW25-20	25ESI20001	GW	N/A	Sample	PFAS	See Work Plan	SEAD 25 Perimeter Well
MW25-21	25ESI20002	GW	N/A	Sample	PFAS	See Work Plan	SEAD 25 Perimeter Well
MW25-22	25ESI20003	GW	N/A	Sample	PFAS	See Work Plan	SEAD 25 Perimeter Well
MW25-23	25ESI20004	GW	N/A	Sample	PFAS	See Work Plan	SEAD 25 Perimeter Well
MW25-24	25ESI20005	GW	N/A	Sample	PFAS	See Work Plan	SEAD 25 Perimeter Well
MW25-25	25ESI20006	GW	N/A	Sample	PFAS	See Work Plan	SEAD 25 Perimeter Well
MWFH-01	25ESI20007	GW	N/A	Sample	PFAS	See Work Plan	Fire House Well
MWFH-02	25ESI20008	GW	N/A	Sample	PFAS	See Work Plan	Fire House Well
MWFH-03	25ESI20009	GW	N/A	Sample	PFAS	See Work Plan	Fire House Well
TBD	25ESI20010	GW	N/A	Duplicate	PFAS	See Work Plan	Duplicate
SEAD 26 Sample Locations and Nomenclature							
MW26-12	26ESI20001	GW	N/A	Sample	PFAS	See Work Plan	Upgradient
MW26-13	26ESI20002	GW	N/A	Sample	PFAS	See Work Plan	Side-Gradient
MW26-14	26ESI20003	GW	N/A	Sample	PFAS	See Work Plan	Side-Gradient
MW26-15	26ESI20004	GW	N/A	Sample	PFAS	See Work Plan	Down-Gradient
MW26-16	26ESI20005	GW	N/A	Sample	PFAS	See Work Plan	Down-Gradient
MW26-17	26ESI20006	GW	N/A	Sample	PFAS	See Work Plan	Down-Gradient
TBD	26ESI20007	GW	N/A	Duplicate	PFAS	See Work Plan	Duplicate
QA/QC Samples							
TBD[2]	25ESI00001 / 26ESI00001	Aqueous	--	Equipment Blank	PFAS	See Work Plan	One equipment blank per day.[2]
TBD[3]	25ESI01000 / 26ESI01000	Aqueous	--	Field Blank	PFAS	See Work Plan	One field blank per day.[3]
TBD[4]	25ESI00100 / 26ESI00100	Aqueous	--	Trip Blank	PFAS	See Work Plan	One trip blank per cooler shipment[4]

Key: TBD = To be determined; GW = groundwater; SA = Sample; DU = Duplicate; MS/MSD = Matrix Spike / Duplicate; EB = Equipment Blank; FB = Field Blank; TB = Trip Blank

(1) MS/MSD and field duplicates will be collected from both SEAD 25/Fire House and SEAD 26 at a rate of 1:20 and 1:10, respectively. The locations of the MS/MSD and field duplicate will be determined in the field based on site conditions. One of the existing sample IDs in the table above will be appended with MS and MSD (e.g., MW25-20MS and MW-25-20MSD). Each set of MS/MSD samples will have a low and moderate spike. The field duplicate will be collected at the same locations as the MS/MSD. Field duplicate sample ID will be one larger than the last ID shown in the table (e.g., MW25-26).

(2) One equipment (rinse) blank will be collected per day of drilling and per day of groundwater sampling. Each day the equipment (rinse) blank ID will increase by one. The equipment blank ID will start with those collected during drilling.
(3) One field blank will be collected per day of groundwater sampling. Each day the field blank ID will increase by one.
(4) One trip blank will be included per cooler of samples shipped. Each trip blank will increase by one.
(5) Secondary well IDs will start one larger increment than the highest primary well ID (e.g., MW25-26 or MWFH-04).
(6) New locations will be drilled so no screen depth is known at this time.

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

(EPA UFP-QAPP Guidance Manual, Section 3.1.1)

This worksheet summarizes the analytical methods for each sampling matrix, including the required sample volume, containers, preservation, and holding time requirements. Details concerning sampling handling are included on **Worksheets #26 & 27**. All samples will be delivered to Katahdin Analytical Services, located in Scarborough, ME (1,4-D samples), TestAmerica Denver (perchlorate samples), or TestAmerica Sacramento (PFAS samples) with ice via UPS or FedEx next day delivery.

Table 19.1 – Sample Containers, Preservation and Hold Times

ANALYTE/ ANALYTICAL GROUP	MATRIX	METHOD/SOP REFERENCE (1)	ACCREDITATION EXPIRATION DATE	CONTAINERS (NUMBER, SIZE, AND TYPE) (2)	PRESERVATION REQUIREMENTS	PREPARATION HOLDING TIME	ANALYTICAL HOLDING TIME	DATA PACKAGE TURNAROUND
SVOCs	GW	3520C, 8270D SIM / CA-213, CA-502	01 Feb 2019	2, 1 liter (L) amber glass bottles	Cool ≤6°C	7 days	40 days	21 days
Perchlorate	GW	6860 / DV-LC-0024	31 Oct 2019	1, 125 ml, HDPE	Cool ≤6°C	28 days	28 days	21 days
Perchlorate	Soil	6860 / DV-LC-0024	31 Oct 2019	1, 4oz., amber glass jar	Cool ≤6°C	28 days	28 days	21 days
PFAS	GW	537_Modified / WS-LC-0025	20 Jan 2021	2, 250 ml HDPE bottles	4 ± 2°C;	14 days	40 days	21 days

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

(2) Sample size is a minimum, the containers listed will be filled to compensate for any required re-analysis or re-extractions. For samples requiring Matrix Spike/Matrix Spike Duplicate (MS/MSD) containers listed should be tripled.

Worksheet #20: Field Quality Control

(EPA UFP-QAPP Guidance Manual, Section 3.1.1)

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

Table 20.1 – Perchlorate Field and Quality Control Samples

SITE	MATRIX	ANALYTICAL GROUP	ESTIMATED NO. OF FIELD SAMPLES	TRIP BLANK (FOR VOC ONLY)	EQUIPMENT BLANK	FIELD DUPLICATES	MATRIX SPIKE / MATRIX SPIKE DUPLICATES	ESTIMATED NUMBER OF TOTAL ANALYSES
OD Grounds	Groundwater	Perchlorate	9	N/A	N/A[1]	10%	5%	12
OD Grounds	Soil		20	N/A	1 per week			24

(1) Dedicated tubing or disposable equipment will be used to collect groundwater therefore equipment blanks are not applicable.

Table 20.2 – 1,4-Dioxane Field and Quality Control Samples

SITE	MATRIX	ANALYTICAL GROUP	ESTIMATED NO. OF FIELD SAMPLES	TRIP BLANK	FIELD BLANK	EQUIPMENT BLANK	FIELD DUPLICATES	MATRIX SPIKE / MATRIX SPIKE DUPLICATES	ESTIMATED NUMBER OF TOTAL ANALYSES
Ash Landfill	Groundwater	1,4-Dioxane	8	N/A	N/A	1 per week	10%	5%	12

Table 20.3 – PFAS Field and Quality Control Samples

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

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

Worksheet #23: Analytical Standard Operating Procedures

(EPA UFP-QAPP Guidance Manual, Section 3.2.1)

The applicable SOPs to be used for analysis of samples collected during the investigation are listed in the below tables. The laboratory SOP references were provided by Katahdin and TestAmerica are presented in **Attachment 1 of this addendum**.

Table 23.1 – Analytical SOPs

SOP #	TITLE, DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX/ ANALYTICAL GROUP	SOP OPTION OR EQUIPMENT TYPE	MODIFIED FOR PROJECT?
CA-213	Analysis of Semivolatile Organic Compounds By: SW 846 Method 8270 – Modified for Selected Ion Monitoring (SIM), 09/17, Revision 14.	Definitive	Groundwater/SVOCs SIM	GC/MS	N
CA-502	Preparation of Aqueous Samples For Extractable Semivolatile Analysis, 09/17, Revision 11.	Definitive	Groundwater/SVOCs SIM	NA	N
DV-LC-0024	Perchlorate in Water and Solids by IC/MS/MS [SW-846 Method 6860], 10/17, Revision 9.	Definitive	Groundwater, Soil/Perchlorate	IC/MS/MS	N
WS-LC-0025	Per- and Polyfluorinated Substances (PFAS) in Water, Soils, Sediments and Tissue by LC/MS/MS, 8/20/18, Revision 3.2	Definitive	Groundwater/PFAS	LC/MS/MS	N

Worksheet #24: Analytical Instrument Calibration

(EPA UFP-QAPP Guidance Manual, Section 3.2.2)

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

Table 24.1 – Analytical Instrument Calibration

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
GC/MS (SIM)	DFTPP Tune	NA	Prior to ICAL and prior to each 12-hour period of sample analysis	Specific ion abundance criteria of BFB from method (Criteria also listed in Section 7.4, current revision of SOP CA-213).	Retune instrument and/or clean source and verify	Analyst, Department Manager	CA-213
GC/MS (SIM)	Performance Checks	NA	At the beginning of each 12-hour period, before analysis of samples DDT breakdown and tailing factors are considered overall measures of port inertness and column performance and are required checks for SIM operation. DDT breakdown and tailing factor checks can be acquired as a full scan.	Degradation ≤20% for DDT	Correct problem, then repeat performance checks. No samples shall be analyzed until the performance checks are within criteria.	Analyst, Department Manager	CA-213
GC/MS (SIM)	Establish Retention Time (RT) Window Position	NA	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	Analyst, Department Manager	CA-213
GC/MS (SIM)	Evaluation of Relative Retention Times (RRTs)	NA	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	CA-213
GC/MS (SIM)	Initial Calibration (ICAL) - Five-point initial calibration	0.25 – 15 ug/ml	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: • Option 1: RSD for each analyte = 20%;	Correct problem then repeat ICAL.	Analyst, Department Manager	CA-213

Table 24.1 – Analytical Instrument Calibration

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
	is required for all SVOCs.			• Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; • Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.			
GC/MS (SIM)	Second Source Calibration or Initial Calibration Verification (ICV)	NA	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	CA-213
GC/MS (SIM)	Continuing Calibration Verification (CCV)	NA	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; t	Analyst, Department Manager	CA-213
IC/MS/MS	Tune Check	NA	Prior to ICAL and after any mass calibration or maintenance is performed.	Tuning standards must span the mass range of the analytes of interest and meet acceptance criteria outlined in the lab SOP.	If the tune check fails, retune instrument and verify. If the tune check will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone. No samples shall be analyzed without an acceptable tune check.	Analyst, Department Manager	DV-LC-0024
IC/MS/MS	ICAL. Seven point initial calibration for target analyte	0.02 – 1.0 µg/L	At instrument set-up, after ICV or CCV failure or after maintenance or major changes such as IC column type	ICAL must meet one of the two options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.995	Instrument and standards are checked. Correct problem. Continue once initial calibration meets criteria.	Analyst, Department Manager	DV-LC-0024
IC/MS/MS)	Second Source Calibration or	NA	Second source standard, immediately	Perchlorate concentration must be within ± 15% of its true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	DV-LC-0024

Table 24.1 – Analytical Instrument Calibration

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
	Initial Calibration Verification (ICV)		following Initial Calibration Blank (ICB) and immediately following ICAL				
IC/MS/MS	Continuing Calibration Verification (CCV)	NA	On days an ICAL is performed, after every 10 field samples, and at the end of the analytical sequence. On days an ICAL is not performed, at the beginning of the sequence, after every 10 field samples, and at the end of the analytical sequence	Perchlorate concentration must be within ± 15% of its true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action and repeat CCV and all associated samples since last successful CCV. Alternatively, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV	Analyst, Department Manager	DV-LC-0024
IC/MS/MS)	Isotope Ratio. 35Cl/37Cl (If tandem MS, this monitors both the parent ion at masses 99/101 and the daughter ion at masses 83/85)	NA	All samples, spiked samples, standards and method blanks	Monitor for either the parent ion at masses 99/101 or the daughter ion at masses 83/85 depending on which ions are quantitated. Must fall within 2.3 – 3.8	If criteria not met, the sample must be rerun. If the sample was not pretreated, the sample must be extracted using cleanup procedures. If after cleanup, the ratio still fails, use alternative techniques to confirm presence of perchlorate, e.g., a post spike sample or dilution to reduce any interference. If acceptance criteria still not met, data must be qualified with a Q-flag and explained in the case narrative. Any procedures used to eliminate the interference must be described in the case narrative	Analyst, Department Manager	DV-LC-0024
IC/MS/MS	Internal Standard	NA	^{18}O-labeled perchlorate must be	Measured ^{18}O IS area must be within + 50% for the average	Rerun sample at increasing dilutions until the criteria	Analyst, Department Manager	DV-LC-0024

Table 24.1 – Analytical Instrument Calibration

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
			added to all field samples, QC samples (batch and instrument) and standards as an internal standard	of the IS area counts of the ICAL and the RRT of the perchlorate ion must be 1.0 ± 2% (0.98-1.02). If peak is not within retention time window, presence is not confirmed	are met. If dilution does not resolve the problem the sample must be reprepped using additional pretreatment steps. If these additional steps fail, apply Q-flag and explain in the case narrative. Flagging is not appropriate for failed standards		
IC/MS/MS)	Interference Check Sample (ICS)	NA	Every Batch of 20 samples and must undergo the same preparation and pretreatment steps as the samples.	80-120%	Check the calibration standards and instrument conditions (may need to replace column). Repeat ICAL 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 sample may be reported that are associated with a failing ICS.	Analyst, Department Manager	DV-LC-0024
IC/MS/MS	Laboratory Reagent Blank (LRB)	NA	Immediately prior to initial calibration and at the end of the analytical sequence	No perchlorate >1/2 the LOQ	Repeat until no carryover and reanalyze samples in associated batch.	Analyst, Department Manager	DV-LC-0024
IC/MS/MS	Interference Threshold Study	NA	At initial setup and when major changes occur in methods operating procedures	Threshold = concentration of common suppressors where perchlorate recovery falls outside 80-120%	NA	Analyst, Department Manager	DV-LC-0024
IC/MS/MS	Mass calibration with PEG or other appropriate material	NA	As needed (failed tune criteria) , after major maintenance, minimum of annually.	+/- 0.5 amu	Recalibrate	Analyst, Department Manager	DV-LC-0024

Table 24.1 – Analytical Instrument Calibration

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
	bracketing mass calibration range						
LC/MS/MS	Mass Calibration	NA	Prior to initial use and after any major maintenance is performed	Calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. Entire range needs to be mass calibrated	NA	Analyst, Department Manager	WS-LC-0025
LC/MS/MS	Tune Check	NA	Prior to ICAL and after any mass calibration or maintenance is performed.	Tuning standard must contain analytes of interest or appropriate substitute. Mass assignments of tuning standard within 0.5 amu of true value by 10:1 S/N for all analytes in the lowest calibration point..	Retune instrument. If the tuning will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Analyst, Department Manager	WS-LC-0025
LC/MS/MS	ICAL- Minimum 5-point initial calibration for target analytes, lowest concentration standard at or below the reporting limit	0.5- 400 ng/mL	Initial calibration prior to sample analysis	S/N ratio > 10:1 for all ions used for quantitation. Confirmation ions for PFOS and PFOA must have S/N > 3:1. The %RSD for all analytes must be <20%. Linear or non-linear calibrations must have r2 > 0.99 for each analyte. Each analyte must be within 70-130% of its true value for each calibration standard.	Evaluate standards, chromatography, and mass spectrometer response. If problem found with above, correct as appropriate, then repeat initial calibration.	Analyst, Department Manager	WS-LC-0025
LC/MS/MS	ICV or Second Source Verification (SSV)	NA	Once per initial calibration, following initial calibration.	All reported analytes and labeled compounds within ±30% of true value	Evaluate data. If problem (e.g., concentrated standard, plugged transfer line) found, correct, then repeat SSV. If it still fails, then repeat initial calibration.	Analyst, Department Manager	WS-LC-0025
LC/MS/MS	CCV	NA	Before sample analysis, after every 10 samples, and at the end of the sequence any mass calibration or maintenance is performed.	All reported analytes and labeled compounds within ±30% of true value	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with case narrative comment. For closing CCVs, if compounds are not identified as critical compounds of	Analyst, Department Manager	WS-LC-0025

Table 24.1 – Analytical Instrument Calibration

INSTRUMENT	CALIBRATION PROCEDURE	CALIBRATION RANGE	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	TITLE/POSITION FOR RESPONSIBLE CORRECTIVE ACTION	SOP REFERENCE
					concern report results with qualifiers. For closing CCVs, if the compound is identified as a critical compound of concern, then recalibrate, and reanalyze all affected samples since the last acceptable CCV; or immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.		
LC/MS/MS	Instrument sensitivity check (ISC)	NA	Prior to analysis and at least once every 12 hours. ISC can serve as a bracketing CCV.	Analyte concentrations must be at the LOQ and within + 30% of their true values.	Correct problem, rerun ISC. If problem persists repeat ICAL.	Analyst, Department Manager	WS-LC-0025

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

(EPA UFP-QAPP Guidance Manual, Section 3.2.3)

This worksheet provides information on analytical instruments and equipment, maintenance, testing, and inspection. To ensure that the analytical instruments and equipment are available and in working order when needed, all laboratory analytical equipment will undergo maintenance and testing procedure in accordance with the laboratory SOPs (provided in **Attachment 1 of this addendum**).

Table 25.1 – Analytical Instrument and Equipment Maintenance, Testing and Inspection

INSTRUMENT/ EQUIPMENT	MAINTENANCE ACTIVITY	TESTING ACTIVITY	INSPECTION ACTIVITY	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION	RESPONSIBLE PERSON	SOP REFERENCE (1)
GC/MS SVOCs (SIM)	Check pressure and gas supply daily. Manual tune if DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	SVOC (SIM)	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-213
IC/MS/MS	Replace columns as needed, change filters and seals, clean lenses and needles, check eluent reservoirs	Perchlorate	Instrument performance and sensitivity	Daily or as needed	Acceptable CCV	Correct problem and recalibrate	Analyst	QAM Section 20
LC/MS/MS	Replace columns as needed, check eluent reservoirs	PFAS	Instrument performance and sensitivity	Daily or as needed	Acceptable CCV	Correct problem and recalibrate	Analyst	WS-LC-0025

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

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

(EPA UFP-QAPP Guidance Manual, Section 3.3)

26.1 SAMPLE NUMBERING

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

Table 26.1 – Sample Numbering Nomenclature

AA = AREA/SITE CODE	ST = STUDY ID	##### = 5 DIGIT NUMERICAL CODE
AL = Ash Landfill	FS = Feasibility Study	000## = Field QC items (e.g., Rinsate Blanks)
45 = SEAD 45 (OD Grounds)	MI = Mini-Investigation	001## = Shipment QC samples (e.g., Trip Blanks)
25 = SEAD-25		1#### = Soil Samples
26 = SEAD-26	--	2#### = Groundwater Samples
	--	3#### = Surface Water Samples
	--	4#### = Sediment Samples

Every sample number will be preceded by the site name designation to identify the site from which the sample was collected. The numerical component for each sample will building upon past LTM events. For database consistency, the next event sample sequence will begin with a sample ID that is one increment higher than the last sample from the previous LTM event. Sample name/numbering examples are shown in **Table 26.1**, and the complete sample list for the next round of sampling for each site is detailed on **Worksheet #18**.

Table 26.2 – Sample Name/Numbering System by Site

SITE	SITE NAME DESIGNATION	EXAMPLE SAMPLE ID (1)
OD Grounds (GW)	45FS	45FS20001
OD Grounds (Soil)	45-FS-SS-#####-Depth 45-FS-SB-#####-Depth	45-FS-SS-10001-0-0.5 45-FS-SB-10001-2.5-4.5
SEAD 25	25ESI	25ESI20001
SEAD 26	26ESI	26ESI20001
Ash Landfill	ALMI	ALMI20001

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

26.2 SAMPLE HANDLING

To ensure sample authenticity and data defensibility, proper sample handing system procedures will be followed from the time of sample collection to final sample disposal. The Contractor Sample Team Lead or designee is responsible for completing the sample bottle label and chain of Custody CoC form, sample collection, sample packing, and coordination of sample shipment. Perchlorate samples will be sent to TestAmerica in Denver, Colorado via FedEx or UPS Next Day Delivery service. 1,4-dioxane samples will be sent to the analytical laboratory, Katahdin in Scarborough, Maine via FedEx or UPS Next Day Delivery service. The PFAS samples will be sent for analytical testing to TestAmerica Laboratories in West Sacramento, California via FedEx or UPS Next Day Delivery service.

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

Table 26.3 – Responsibilities for Sample Handling, Custody, and Disposal

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

26.2.1 Sample Labeling

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

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

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

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

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

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

TestAmerica Denver
4955 Yarrow Street
Arvada, CO 80002
Tel.: (303) 736-0100

The following address will be used for 1,4-dioxane sample shipments:

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

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

All laboratory sample receipt, internal custody and sample archiving, and disposal procedures shall be completed in accordance with Katahdin SOPs: SD-902-11 and SD-903-05, TestAmerica Denver SOPs: DV-HS-0005 and DV-QA-0003, and TestAmerica West Sacramento SOPs: WS-QA-0003, WS-QA-0034, and WS-EHS-0001.

26.3.2 SAMPLE IDENTIFICATION PROCEDURES

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

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

26.3.3 CHAIN-OF-CUSTODY PROCEDURES

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

26.3.4 NON-CONFORMANCE

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

Worksheet #28: Analytical Quality Control and Corrective Action

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

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

28.1 SVOCS BY EPA SW-846 METHOD 8270D SIM IN GROUNDWATER

Matrix: Groundwater
Analytical Group: SVOCs (1,4-dioxane)
Analytical Method: EPA SW-846 Method 8270D SIM
SOP: CA-213

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

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT-SPECIFIC MPC
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprepare and reanalyze MB and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Surrogate	Included in every field sample, standard, blank, and QC sample	Laboratory derived limits: 1,4-Dioxane-d8 = 30-115%.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparation batch of twenty or fewer samples of similar matrix.	A laboratory must use the QSM Appendix B Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. (See Table 28.1a below)	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT-SPECIFIC MPC
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	As specified on the chain-of-custody by Parsons	A laboratory must use the QSM Appendix B Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. (See Table 28.1a below). RPD of all analytes ≤ 40% (between MS and MSD	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Internal Standard (IS)	Included in every field sample, standard, blank and QC sample. (1,4-dichlorobenzene-d4)	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	Not applicable (NA)	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). If outside acceptance criteria, the parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.	Parsons Data Validator or Project Chemist	See Worksheet #12
Equipment Blank (EB)	1 per week for SEAD 25, SEAD 45, and Ash Landfill sites. NA for all other sites	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12

Table 28.1a - LCS/MS/MSD Control Limits for SVOCs in Groundwater by SW8270D SIM

COMPOUNDS	LCS/MS/MSD CONTROL LIMITS (%R)
1,4-Dioxane	26-103
1,4-Dioxane-d8 (Surrogate)	30-115

28.2 PERCHLORATE IN GROUNDWATER AND SOIL EPA SW-846 METHOD 6860

Matrix: Groundwater and Soil
Analytical Group: Perchlorate
Analytical Method: EPA SW-846 Method 6860
SOP: DV-LC-0024

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

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Method Blank	One per preparation batch of 20 or fewer samples.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank. If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative.	Analyst, Department Supervisor	No Target Compounds >1/2 LOQ. Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of up to 20 samples.	The laboratory must use the QSM Appendix B Limits for batch control. (See table 28.2a and 28.2b below)	If the LCS recovery is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. If insufficient sample or corrective action fails, then apply Q-flag to specific analyte(s) in all samples in the associated prep batch. Must be explained in the case narrative	Analyst, Department Supervisor	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One MS/MSD per 20 field samples	A laboratory must use the QSM Appendix B Limits for batch control. (See Table 28.2a and 28.2b below). RPD of all analytes = ≤15% (between MS and MSD)	Examine the project- specific requirements. Contact the client as to additional measures to be taken.	Analyst, Department Manager	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). If outside acceptance criteria, the parent sample and field duplicate sample will be	Parsons Data Validator or Project Chemist	See Worksheet #12

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
			qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.		
Equipment Blank (EB)	1 per week for SEAD 25, SEAD 45, and Ash Landfill sites. NA for all other sites	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12

Table 28.2a - LCS/MS/MSD Control Limits for Perchlorate in Groundwater by SW6860

COMPOUNDS	LCS/MS/MSD CONTROL LIMITS (%R)
Perchlorate	84-119

Table 28.2b - LCS/MS/MSD Control Limits for Perchlorate in Soil by SW6860

COMPOUNDS	LCS/MS/MSD CONTROL LIMITS (%R)
Perchlorate	84-121

28.3 PFAS IN GROUNDWATER BY EPA METHOD 537 MODIFIED

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

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

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
Method Blank	One per preparatory batch.	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
Internal Standards (Isotope Dilution Analytes, spiked prior to extraction)	Every sample, spiked sample, standard, and method blank	% recovery for each IS in the original sample (prior to dilutions) must be within 50-150%	If recoveries are acceptable for QC samples, but not field samples, the field samples must be re-prepped and reanalyzed (greater dilution may be needed). If recoveries are unacceptable for QC samples, correct problem, and reanalyze all associated failed field samples.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch.	Statistically derived in-house laboratory limits seen in Table 28.9b.	Reanalyze LCS once. If acceptable, report. Evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections for critical chemicals of concern, evaluate and reprep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
MS	One per preparatory batch.	Statistically derived in-house laboratory limits seen in Table 28.3b.	Examine project specific requirements. Contact the client as to additional measures to be taken. Evaluate the data and re-prepare/reanalyze the native sample and MS/MSD pair if laboratory error is indicated. If criteria are not met then explain in the Case Narrative.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.

QC SAMPLE	NUMBER/ FREQUENCY	METHOD/SOP ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	PERSON(S) RESPONSIBLE FOR CA	PROJECT SPECIFIC MPC
MSD	One per preparatory batch.	RPD ≤ 30%.	Evaluate the data and re-prepare/reanalyze the native sample and MS/MSD pair if laboratory error is indicated.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
Post spike Sample	Only applies to aqueous samples prepared by serial dilution instead of SPE that have reported values of <LOQ for analyte(s).	Spike aliquot(s) of sample at the final dilution(s) reported for sample with all analytes that have reported values <LOQ in the final dilution. The spike must be at the LOQ to be reported with the sample. Spike recovery within 70-130% of its true value.	When analyte concentrations are <LOQ, and the recovery does not meet 70-130%, the sample, sample duplicate and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.	Analyst, Laboratory Department Manager	Same as Method/SOP QC Acceptance Limits.
Field duplicate (FD)	1 per 10 field samples collected	See Worksheet #12	NA for Laboratory. Parsons project chemist will discuss with field personnel if necessary (i.e. if a trend is noticed). The parent sample and field duplicate sample will be qualified as estimated and flagged "J" by the data validator when both sample results are ≥ to the LOQ.	Parsons Data Validator or Project Chemist	See Worksheet #12
Equipment Blank (EB)	1 per day from groundwater sampling equipment 1 per day from drilling equipment	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12
Field Blank (FB)	1 per day	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12
Trip Blank (TB)	1 per cooler that includes PFAS samples	See Worksheet #12	NA for laboratory Parsons project chemist will discuss with field personnel or the laboratory if necessary (i.e. if a trend is noticed). The associated field sample results will be qualified/flagged "B" if the result is <5x lab non-common contaminant or <10x lab common contaminant.	Parsons Data Validator or Project Chemist	See Worksheet #12.

Table 28.3b – LCS/MS/MSD Control Limits for Analysis of PFAS in Groundwater

PFAS	LCS/MS/MSD CONTROL LIMITS (%R)
Perfluorobutanoic acid (PFBA)	83-118
Perfluoropentanoic acid (PFPeA)	83-108
Perfluorohexanoic acid (PFHxA)	83-109
Perfluoroheptanoic acid (PFHpA)	80-113
Perfluorooctanoic acid (PFOA)	80-107
Perfluorononanoic acid (PFNA)	83-113
Perfluorodecanoic acid (PFDA)	85-113
Perfluoroundecanoic acid (PFUnA)	76-105
Perfluorododecanoic acid (PFDoA)	87-116
Perfluorotridecanoic Acid (PFTriA)	75-129
Perfluorobutanesulfonic acid (PFBS)	87-120
Perfluorohexanesulfonic acid (PFHxS)	81-106
Perfluoroheptanesulfonic Acid (PFHpS)	80-117
Perfluorooctanesulfonic acid (PFOS)	82-112
Perfluorodecanesulfonic acid (PFDS)	81-114
Perfluorooctane Sulfonamide (PFOSA)	85-114
N-methyl perfluorooctane sulfonamidoacetic acid (NMeFOSAA)	82-111
N-ethyl perfluorooctane sulfonamidoacetic acid (NEtFOSAA)	80-109
6:2 FTS	75-118
8:2 FTS	83-111
Perfluorotetradecanoic acid (PFTeA)	82-115

Worksheet #29: Project Documents and Records

(EPA UFP-QAPP Guidance Manual, Section 3.5.1)

29.1 PROJECT DOCUMENT AND RECORDS

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

Table 29.1 – Laboratory Records (TestAmerica- W. Sacramento and Denver)[1]

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

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

Worksheet #36: Data Validation Procedures

(EPA UFP-QAPP Guidance Manual, Section 5.2.2)

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

36.1 VALIDATION PROCESS

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

Table 36.1 - Overview of Analytical Data Validation

DATA VALIDATORS: PARSONS	
Analytical Group/Method:	All Chemical Analyses
Data deliverable requirements:	Level IV data packages and EDDs (NYSDEC EDD for PFAS and 1,4-D data)
Analytical specifications:	Per UFP-QAPP, DoD QSM version 5.1 (specific to PFAS, Appendix B, Table 15) and DoD QSM version 5.1 (specific to Perchlorate and SVOCs by method 8270D SIM), Katahdin and TA Denver SOPs
Measurement performance criteria:	Per UFP-QAPP and DoD QSM version 5.1 (specific to PFAS, Appendix B, Table 15) and DoD QSM version 5.1 (specific to Perchlorate and SVOCs by method 8270D SIM)
Percent of data packages to be validated:	100%- Level IV data validation as described in SOP CHEM-01
Percent of raw data reviewed:	100%
Percent of results to be recalculated:	10%
Validation procedure:	Per UFP-QAPP, DoD QSM version 5.1 (specific to PFAS, Appendix B, Table 15) and DoD QSM version 5.1 (specific to Perchlorate and SVOCs by method 8270D SIM)
Data validation codes:	See table below
Electronic validation program/version:	Excel CSV file or NYSDEC EDD format

Table 36.2 - Data Validation Codes and Definitions

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

Note: Labs will report all concentration down to Detection Limit (DL) and flag any results between DL and LOQ with "J". All non-detected will be reported as <LOD, per DoD QSM version 5.1.

References

EPA, 1998. Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater. September 1998.

EPA, 2016. Environmental Protection Agency. Table of Regulated Drinking Water Contaminants. July 2016. https://www.epa.gov/ground-water-and-drinking-water/table-regulated-drinking-water-contaminants

EPA, 2018. Regional Screening Level (RSL) Summary Table (TR=1E-06, HQ=0.1). May 2018. https://semspub.epa.gov/work/HQ/197235.pdf

Intergovernmental Data Quality Task Force, 2012. Uniform Federal Policy for Quality Assurance Project Plans. Optimized UFP-QAPP Worksheets. March 2012. https://www.epa.gov/sites/production/files/documents/ufp_qapp_worksheets.pdf

NYSDEC, 1998. New York Department of Environmental Conservation. Division of Water Technical and Operational Guidance Series (1.1.1). June 1998. http://www.dec.ny.gov/docs/water_pdf/togs111.pdf

Parsons Engineering Science, Inc., 1995. Expanded Site Inspection (ESI) – Seven High Priority SWMUs SEADs 4, 16, 17, 24, 25, 26, and 45. December 1995.

Parsons, 2004. Record of Decision for the Ash Landfill Operable Unit, Final, July 2004.

Parsons, 2006a. Final Sampling and Analysis Plan for Seneca Army Depot Activity (SAP), October 2006.

Parsons, 2006b. Remedial Design Work Plan for the Ash Landfill Site at Seneca Army Depot Activity, July 2006.

Parsons, 2006c. Remedial Design Report for the Ash Landfill Operable Unit, August 2006.

Parsons, 2016c. Annual Report and Year 9 Review for the Ash Landfill Operable Unit. Seneca Army Depot Activity. September 2016.

Parsons, 2018a. Final (Revision 1) Feasibility Study Report for Open Detonation Grounds Munitions Response Action, Seneca Army Depot Activity, Romulus, New York. In progress.

Parsons, 2018b. Final 2017 PFAS Site Inspection Report. SEAD 25 (Fire Training and Demonstration Pad), SEAD 26 (Fire Training Pit and Area), SEAD 122E (Airfield and Refueling Pads), Seneca Army Depot Activity. January 2018.

Parsons, 2018c. Final Perchlorate Soil and Groundwater Investigation Work Plan. Seneca Army Depot Activity. August 2018.

Parsons, 2018d. Draft 1,4-Dioxane Groundwater Investigation Work Plan, Seneca Army Depot Activity, August 2018.

Parsons, 2018e. Draft Work Plan for the PFAS Expanded Site Investigation, SEAD 25 and SEAD 26, Seneca Army Depot Activity. October 2018.

US Army Environmental Hygiene Agency (USAEHA), 1987. Interim Final Report, Groundwater Contamination Survey No. 38-26-0868-88, July 1987.

Appendices

APPENDIX A – CONTRACTOR SOPS

APPENDIX B – ANALYTICAL SOPS

APPENDIX C – HISTORICAL REPORTS

APPENDIX D – RESUMES

APPENDIX E – RESPONSE TO COMMENTS

Appendix A

Contractor SOPs

Additional SOPs provided which are not found in the Final UFP-QAPP. Reference PFAS Work Plan for PFAS-specific SOPs.

Procedure # ENV-01	Title: SOIL SAMPLING	Revision # 03
Effective Date: 03/11/15	**Approved By:** Thomas Mills, PG	**Last Reviewed/Revised:** 10/07/15

1. PURPOSE

The purpose of this SOP is to describe the general methods to be employed when collecting surface or subsurface soil samples for analysis during munitions response projects. Types of surface soil samples may include discrete, seven-point wheel, incremental sampling method (ISM), and/or Terra Core® samples. Subsurface soil samples may be collected using hand augers or direct push methods (e.g. Geoprobe®). This procedure also applies to the collection of dry sediment samples.

2. RESPONSIBILITIES

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

3. RELEVANT DEFINITIONS

Term	Definition
None	Not applicable.

4. REQUIRED EQUIPMENT

Equipment	Brief Description of Function and Purpose
Sampling tools	A stainless steel or disposable spoon/trowel/ or scoop (s), incremental sampling method (ISM) sampling tool(s), hand augers, etc. for sample collection.
Sample containers	Jars, bottles, or pre-cleaned bags as specified in the approved work plan for sample containerization. Coolers for sample shipment.
Logbook	For documenting sampling activities.
GPS Unit	To record sample coordinates.
Chain-of-custody (CoC) forms	For tracking sample details and chain-of-custody, and for providing instruction on sample analysis to analytical laboratory.

Procedure # ENV-01	Title: SOIL SAMPLING	Revision # 03
Effective Date: 03/11/15	**Approved By:** Thomas Mills, PG	**Last Reviewed/Revised:** 10/07/15

5. PROCEDURE

5.1. Health and Safety

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

5.2. General Requirements for all Sample Methods

5.2.1. Documentation

The Sampling Team Leader or designee shall record the description of sample locations, soil type, and any other relevant or notable details in the Field Sampling Logbook and/or on project-specific sampling forms. The Sampling Team Leader or designee shall also record the sample locations using a global positioning system (GPS) unit (e.g., Trimble® GeoXT™ or similar) and document sample coordinates in the Field Sampling Logbook. The Sampling Team Leader or designee shall record other information as specified in the approved work plan, including completion of a Daily Quality Control (QC) Report (DQCR) in accordance with **SOP ENV-00, Daily Quality Control Report**.

5.2.2. Sampling Handling and Shipment

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

5.2.3. Sample Analysis and Quality Control Samples

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

5.2.4. Anomaly Avoidance

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

Procedure # ENV-01	Title: SOIL SAMPLING	Revision # 03
Effective Date: 03/11/15	**Approved By:** Thomas Mills, PG	**Last Reviewed/Revised:** 10/07/15

5.3. Sampling Methods for Surface Soil

5.3.1. Preparation for Surface Soil Sampling

The following steps shall be completed when preparing for collection of surface soil samples:

1. The Sampling Team Leader shall review the applicable section(s) of the work plan to confirm the sample location, quantities, required sample containers, and other relevant information.
2. The Sampling Team will navigate to the sample location, make initial observations, and complete the required documentation (see Section 5.2.1).
3. If MEC hazards are present, the MEC Escort shall practice anomaly avoidance (see Section 5.2.4).
4. The Sampling Team shall don clean gloves before each sampling event.
5. The Sampling Team shall assemble the necessary sampling equipment and supplies, sample containers, decontamination materials, etc in the sampling area.

5.3.2. Discrete Sampling Method for Soil (or Dry Sediment)

5.3.2.1. The discrete sampling method is best suited to identifying localized contamination. Discrete sampling may be used to collect a sample from a biased area of soil (e.g. stained soil, underneath observed MEC/munitions debris, the bottom of an excavation). Discrete sampling is also used when collecting IDW samples from drums or spoils piles to characterize waste.

5.3.2.2. Following the preparatory actions (Section 5.3.1), the Sampling Team shall complete the following steps to collect discrete surface soil samples:

1. Collect the sample using an approved sampling tool (e.g., stainless steel or disposable spoon, trowel, or scoop).
2. Transfer the collected soil from the sample tool directly into the sample container(s).
3. When sample containers are filled, secure the caps tightly on the containers and place on ice as soon as possible (if required by sample preservation method).
4. After sampling is completed, backfill the hole with remaining soil to return the site to as close to original condition as possible.
5. Perform post-sampling activities (Section 5.3.6).

5.3.3. Seven-point Wheel Method for Soil (or Dry Sediment)

5.3.3.1. The seven-point wheel composite sampling method (*also referred to as the Cold Regions Research and Engineering Laboratory's [CRREL] Seven-point Wheel Sampling Approach*) is used to collect composite soil samples. Composite sampling is generally used to characterize the immediate vicinity of a chosen location (e.g., a detonation crater).

5.3.3.2. Following the preparatory actions (Section 5.3.1), the Sampling Team shall complete the following steps to collect seven-point wheel surface soil samples:

1. Prepare an approved sampling tool (e.g., stainless steel or disposable spoon, trowel, or scoop).
2. Collect seven small sub-samples from an approximately 4-foot diameter area. Collect six of the sub-samples at evenly spaced intervals around the circumference of the circle and one sub-sample in the center of the circle (**Exhibit 1**).
3. Place the seven sub-samples into a large disposable or stainless steel bowl and mix the combined soil thoroughly to ensure a representative sample.
4. Transfer the mixed soil into the sample container(s). When sample containers are filled, secure the caps tightly on the containers and place on ice as soon as possible (if required by sample preservation method).

Procedure # ENV-01	Title: SOIL SAMPLING	Revision # 03
Effective Date: 03/11/15	**Approved By:** Thomas Mills, PG	**Last Reviewed/Revised:** 10/07/15

5. After sampling is completed, backfill the hole with remaining soil to return the site to as close to original condition as possible.
6. Perform post-sampling activities (Section 5.3.6).

5.3.4. Incremental Sampling Method for Soil (or Dry Sediment)

5.3.4.1. ISM samples are collected to evaluate contamination over larger areas. ISM defines one or more sampling units (SU) to be sampled at a site, from which multiple sample "increments" are then collected and composited into a single, representative incremental sample from each SU. The purpose of ISM is to provide a more accurate measure of the mean concentration of contaminants in each SU by providing reproducible, scientifically defensible data. Surface soil samples collected using the ISM will follow the *ITRC Incremental Sampling Methodology Technical and Regulatory Guidance*, published by ITRC in February 2012. **Note**: *ISM samples are best suited to evaluating inorganic and non-volatile organic analytes. ISM samples will not be used to evaluate chemical agent or agent breakdown products*.

5.3.4.2. In addition to the standard preparatory actions (Section 5.3.1), the following actions also need to be completed to prepare for collecting ISM samples:

1. Identify SU Size(s) and Location(s): The size and location of SUs, sample depth, and the required number of sample increments, are specified in the approved work plan. Note that for ISM samples, only the SU corner locations need to be recorded using GPS; the locations of individual sample increments do not need to be recorded.
2. Determine Increment Grid Configuration and Interval: Once the SU has been designated, the Sampling Team Leader or designee will determine the approximate configuration and interval(s) at which sample increments will be collected. These will be designed to be regularly spaced throughout the collection grid to the extent possible. For example, if 30 sample increments are required, five increments should be collected along six rows (i.e., a 5 x 6 sampling grid). If the SU was 100 feet by 100 feet, then the sample spacing would be approximately 20 feet and the rows would be approximately 17 feet apart. Note these distances do not need to be exact, but the Sampling Team shall attempt to collect regularly spaced samples. The sampling interval(s) for each SU shall be recorded by the Sampling Team Leader or designee. An example 10 x 10 sampling grid for an ISM sample is shown in **Exhibit 2**.
3. Select Sample Collection Origin. Once the sample increment interval has been determined, the Sampling Team will randomly select the starting point for sample collection and begin the collection of individual sample increments from each grid cell using the determined sample increment interval. The Sampling Team will endeavor to collect each sample increment from the same relative location in each cell of the sampling grid (see **Exhibit 2**).

5.3.4.3. Following the preparatory actions (Sections 5.3.1 and 5.3.4.2), the Sampling Team shall complete the following steps to collect ISM surface soil samples:

1. Prepare an approved sampling tool (e.g., stainless steel sample corer designed not to discriminate in size, shape or concentration of particles collected for the range of particle sizes of interest).
2. Collect sample increments from the SU as specified in the work plan using the same sampling tool for each increment. Sample increments shall be collected in an unbiased and uniform manner throughout the SU, with each increment having the same size and mass to the greatest extent practicable. Increments shall be combined into a single certified, pre-cleaned plastic bag. These additional measures shall be observed during sampling:

   (a) Vegetated areas will not be avoided and vegetation shall not be removed from sample increment locations where possible.

Procedure # ENV-01	Title: SOIL SAMPLING	Revision # 03
Effective Date: 03/11/15	**Approved By:** Thomas Mills, PG	**Last Reviewed/Revised:** 10/07/15

(b) If pieces of explosive residue (raw high explosives) are present at the sample location, the MEC Escort shall move them prior to sampling, and their location shall be documented.
(c) If a sample increment cannot be collected from a particular location (e.g., rock refusal), then a second increment will be collected next to the original location and documented in the Field Sampling Logbook.
(d) The total weight of all sample increments in each incremental sample will be at least 1 kilogram but no more than 2 kilograms.

3. Once the sample is collected, weigh, label and seal the certified plastic bag. Place on ice as soon as possible (if required by sample preservation method).
4. Perform post-sampling activities (Section 5.3.6).

5.3.5. Terra Core® Sampling Method for Soil (or Dry Sediment)

5.3.5.1. Samples requiring VOC analysis may be collected using EnNovative Technologies Terra Core® samplers. Terra Core® samplers limit the amount of volatilization that occurs during sampling, which allows for a more accurate and valid analytical result.

5.3.5.2. Following the preparatory actions (Section 5.3.1), the Sampling Team shall complete the following steps to collect Terra Core® surface soil samples:

1. Prepare a Terra Core® sampler, and a 40mL VOA vial containing the proper preservative (deionized [DI] water or methanol) and a magnetic stirring bar (if required).
2. With the plunger seated in the handle, push the Terra Core® sampler into the soil until the sample chamber is filled. Wipe all soil or debris from the outside of the Terra Core® sampler. *The soil plug should be flush with the mouth of the sampler*.
3. Rotate the plunger that was seated in the handle to 90° until it is aligned with the slots in the sampler body. Place the mouth of the sampler into the 40mL VOA vial and extrude the sample into the container by pushing the plunger down.
4. Quickly replace the lid of the 40mL VOA vial. *When capping the VOA vial, be sure to remove any soil or debris from the top or threads of the vial*. Place the collected sample on ice as soon as possible (if required by sample preservation method).
5. Perform post-sampling activities (Section 5.3.6).

5.3.6. Post Sampling Activities for Surface Soil Sampling

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

1. The Sampling Team Leader or designee shall label each sample container with the Sample ID, date, time, analysis, and other information required on the sample label.
2. The Sampling Team Leader or designee will confirm the required samples have been collected, including necessary QC samples as specified in the approved work plan.
3. The Sampling Team Leader or designee shall record the sample location GPS coordinates.
4. The Sampling Team will decontaminate reusable sampling equipment as described in Section 5.5 or as specified in the approved work plan.
5. The Sampling Team Leader or designee shall complete the CoC and other required documentation (see Section 5.2.1) and prepare the sample for shipment (see Section 5.2.2).

5.4. Sampling Method for Subsurface Soil

5.4.1. Preparation for Subsurface Soil Sampling

The following steps shall be completed when preparing for collection of subsurface soil samples:

Procedure # ENV-01	Title: SOIL SAMPLING	Revision # 03
Effective Date: 03/11/15	**Approved By:** Thomas Mills, PG	**Last Reviewed/Revised:** 10/07/15

1. The Sampling Team Leader shall review the applicable section(s) of the work plan to confirm the sample location, quantities, required sample containers, and other relevant information.
2. The Sampling Team Leader will obtain any necessary excavation permits and, if necessary, contact a local underground utility locating service to perform a utility clearance for all subsurface sample locations.
3. The Sampling Team will navigate to the sample location, make initial observations, and complete the required documentation (see Section 5.2.1).
4. If MEC hazards are present, the MEC Escort shall practice anomaly avoidance (see Section 5.2.4).
5. The Sampling Team shall don clean gloves before each sampling event.
6. The Sampling Team shall assemble the necessary sampling equipment and supplies, sample containers, decontamination materials, etc in the sampling area. If on-site decontamination is required, arrange the necessary supplies in a nearby but separate location, away from the borehole. All equipment entering the borehole shall be decontaminated.
7. The Sampling Team shall calibrate required equipment and document the calibration on an equipment calibration form.

5.4.2. Direct Push or Hand Auger Method for Subsurface Soil

5.4.2.1. This section provides procedures for subsurface soil sampling using a direct push type rig (e.g., Geoprobe®) or hand auger. If a direct push rig is used, it shall be operated by an appropriately licensed driller.

5.4.2.2. Prior to the advancement of any equipment into a borehole, down hole anomaly avoidance will be conducted in accordance with **SOP MEC-03, MEC Avoidance and Escort**. If a subsurface anomaly is detected during augering or drilling, the borehole will be terminated for safety reasons, the detection depth and location will be noted in the field log, and a sample will be collected at the termination depth.

5.4.2.3. Following the preparatory actions (Section 5.4.1), the Sampling Team shall complete the following steps to collect soil samples from the soil borings advanced by hand augering or direct push rig:

1. Spread clean plastic sheeting on the ground or table at each sampling location to keep sampling equipment clean and prevent cross-contamination.
2. Advance the hand auger or direct push tool to the desired sample depth.
3. Collect the sample using an approved sampling tool (e.g., stainless steel or disposable spoon, trowel, or scoop) and scoop the soil from the auger bucket or acetate liner from the direct push rig starting at representative depth ranges as detailed in the work plan. For hand augering, use a new, clean auger bucket once the top of the sampling depth is reached.
4. Transfer the sample from the auger bucket or trowel into a large disposable or stainless steel bowl and mix the combined soil thoroughly to ensure a representative sample.
   EXCEPTION: If collecting subsurface samples for VOC analysis, the sample will be collected directly from the sample equipment (e.g., auger bucket or acetate sleeve) using a Terra Core® sampler as described in Section 5.3.5. The soil shall not be mixed before sample collection.
5. Collect suitable quantities with the approved sampling tool and transfer directly into the sample container(s).
6. Repeat these steps as necessary to obtain sufficient sample volume.
7. When sample containers are filled, secure the caps tightly on the containers and place on ice as soon as possible (if required by sample preservation method).
8. After sampling is completed, backfill the hole with remaining soil to return the site to as close to original condition as possible.
9. Perform post-sampling activities (Section 5.4.3).

Procedure # ENV-01	Title: SOIL SAMPLING	Revision # 03
Effective Date: 03/11/15	**Approved By:** Thomas Mills, PG	**Last Reviewed/Revised:** 10/07/15

5.4.3. Post Sampling Activities for Subsurface Soil Sampling

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

1. The Sampling Team Leader or designee shall label each sample container with the Sample ID, date, time, analysis, and other information required on the sample label.
2. The Sampling Team Leader or designee will confirm the required samples have been collected, including necessary QC samples as specified in the approved work plan.
3. The Sampling Team Leader or designee shall record the sample location GPS coordinates. *Note that for ISM samples, only the center point of the SU needs to be recorded using GPS; the locations of individual sample increments do not need to be recorded*.
4. The Sampling Team will decontaminate reusable sampling equipment as described in Section 5.5 or as specified in the approved work plan.
5. The Sampling Team Leader or designee shall complete the CoC and other required documentation (see Section 5.2.1) and prepare the sample for shipment (see Section 5.2.2).

5.5. Sampling Equipment Decontamination

5.5.1 Disposable equipment shall be used wherever possible to limit the potential of cross-contamination. However, If reusable equipment is used (e.g. stainless steel bowls and spoons, direct push tooling or cutting shoes) decontamination shall be performed.

5.5.2 Sampling equipment decontamination shall be conducted in an uncontaminated area free of dust. Unless otherwise specified in the approved work plan, sampling equipment will be decontaminated using the following process:

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

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

6. REFERENCES

Reference Title (Author)	Brief summary of relevance to this procedure
Incremental Sampling Methodology, ITRC Technical and Regulatory Guidance (ITRC, 2012)	Guidelines discussing reasoning and procedure for incremental sampling.

Procedure # ENV-01	Title: SOIL SAMPLING	Revision # 03
Effective Date: 03/11/15	**Approved By:** Thomas Mills, PG	**Last Reviewed/Revised:** 10/07/15

7. EXHIBITS

EXHIBIT 1
CRREL 7-POINT WHEEL DIAGRAM

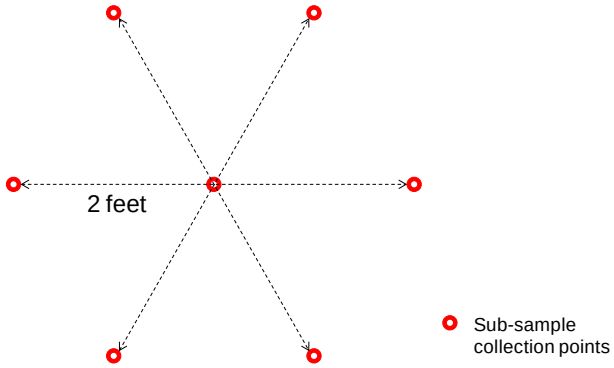
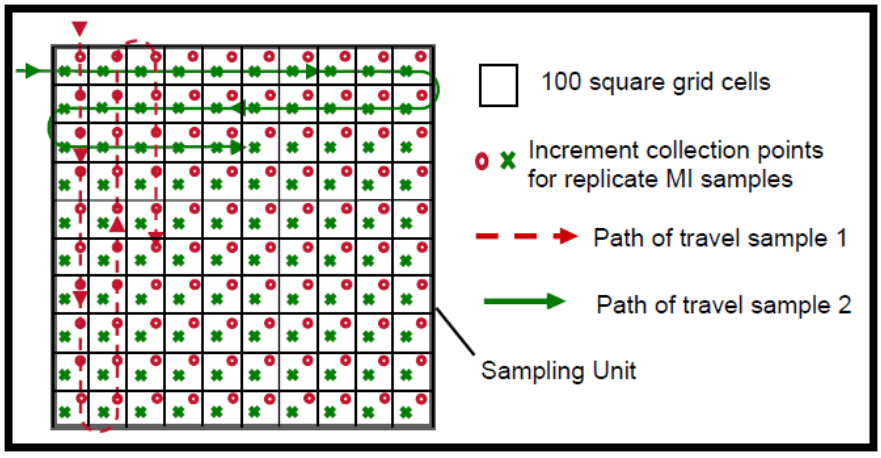


EXHIBIT 2
INCREMENTAL SAMPLING EXAMPLE (USACE, 2009)



Procedure # ENV-01	Title: SOIL SAMPLING	Revision # 03
Effective Date: 03/11/15	**Approved By:** Thomas Mills, PG	**Last Reviewed/Revised:** 10/07/15

8. REVISION HISTORY

Rev.	Date	Summary of Changes	Reason for Revision
00	02/18/15	Initial Release	n/a
01	03/11/15	Minor revisions to ISM text	Scheduled review
02	04/30/15	Ruled out ISM for chemical agent sampling	External comments
03	10/07/15	Added reference to Terra Core® sampling for subsurface soil samples.	External comments

Appendix B

Analytical SOPs

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

Additional SOPs provided which are not found in the Final UFP-QAPP.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: [signature] Date: 020101

Operations Manager: [signature] Date: 1/31/01

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: [signature] Date: 2/07/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	DN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to section 8 & Table 1 to comply with Navy.	DN	09.30.04	09.30.04
03 8270C	Sect. 7.2: Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all references to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. Changed linear regression correlation coefficient criteria. Added MI SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited section 7.5.1 - initial cal. table	LAD	02/08	02/08

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Section 5.3.2.3 - Added calibration Mix B. Section 7.5.1 - Edited to address diffent SIM compounds may need to be calibrated at different levels depending on the compound and project requirements.	LAD	04/09	04/09
07	Changes made for compliance with DoD QSM version 4.1	LAD	08/09	08/09
08	Updated Standard prep. Added compounds to Table 3 and 5, Updated references. Added DoD QSM QC requirements Table.	LAD	04/10	04/10
09	Sect. 7.4 - Added additional tune information. Sect. 7.6 - Added 100 ul minimum extract vol. & 1 ul IS is added for each 100 ul aliquot. Sect. 7.5.4 - Added RRT information Sect. 9.0 - Added MDL, LOD and LOQ information. Table 4 - Added 1,4-Dioxane-d6 Surrogate	LAD	05/11	05/11
10	Sect. 7 - Changed sample volume from 1 ul to 2 ul. Sect. 8 - Added 10% rule for non-DoD clients. Sect. 9 - Added MDL, LOD and LOQ information. Sect. 10 - Added and updated references. Updated Figure 1. Added Addendum 1 - low level 1,4-Dioxane analysis	LAD	05/12	05/12
11	Sect. 1 and 7 - Removed Quickform reporting and added KIMS. Sect. 8 and Table 1 - Added the surrogate 1,4-Dioxane-d8. Throughout - Fixed typos and made minor changes.	LAD	03/13	03/13
12	Sect. 4 - Updated instrument and column models. Sect. 7 - updated calibration levels and prep. Sect. 8 - Added marginal exceedance criteria. Updated ms/msd acceptance criteria. Tables - Added DoD QSM 5.0 QC Requirements. Updated Fig. 2 & 3	LAD	04/14	04/14
13	Sect. 5 - Added Standards to title. Sect. 7 & Appendix 1 - Updated GC/ms operating conditions. Appendix 1. Corrected 1,4 dioxane primary and secondary ions. Add 1,4 dioxane-d4 ions. Changed KAS INC to KAS throughout	LAD	03/16	03/16

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
14	Sect. 10-Updated method references. Sect 7 -Added percent error calculation and relative std error calculation. Sect 1.1-updated definitions. Sect. 9.0-update. Sect. 7-changed the initial cal. std concentrations. Added compounds to Tables 5 & 7.	VAD	09/17	09/17

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

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

I acknowledge receipt of copy ___ of document SOP CA-213-14, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-213-14, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions

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

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

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

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

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, muffled sand is used as a blank matrix. The blank is taken through the appropriate steps of the process. Refer to section 8 for Method Blank acceptance criteria

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

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

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. Note: For the purposes of this SOP, LLOQs, LOQs and PQLs are considered equal terms. The laboratory may use the terms interchangeably. The term, LOQ, must be used for DoD work. Refer to section 9 for specific LOQ/LLOQ verification requirements

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

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

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

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also

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

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

1.3 Safety

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

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

1.4 Pollution Prevention/Waste Disposal

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

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer

to Katahdin SOP CA-502, "Preparation of Aqueous Samples for Extractable Semivolatile Analyses", SOP CA-512, "Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 6890.

4.2 Mass Spectrometers (MS): HP5975 or HP5973

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

4.4 Autosamplers: HP 7673As

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: RTX5 SIL MS - 30m, 0.25mm I.D., 25um film thickness, columns (Restek) or equivalent.

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

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

5.0 REAGENTS AND STANDARDS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer’s expiration date is before this one year date, the manufacturer’s expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 The standards are prepared on an as needed basis (or every 6 months) and stored in screw-cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.2 Calibration Mix A – Prepare standards in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 20 ug/mL.

5.3.2.3 Calibration Mix B - Some compounds must be calibrated at higher concentrations. For these compounds a secondary standard is prepared which will “boost” the concentration of these compounds in the initial calibration. The concentration of this standard is determined on a project to project basis.

5.3.2.4 Internal Standard Solution – Prepare standard in methylene chloride containing 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 at a final concentration of 80 ug/mL.

5.3.2.5 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

5.3.2.6 Independent Calibration Verification (ICV) Standard – From a source independent of the calibration standards, prepare a standard in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 2 ug/mL.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

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

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA
Tune file: DFTPP.U

Method files: LSPSIMXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
L = instrument ID (Each instrument is given a unique identifier)
DFTPP390.M (DFTPP tuning acquisition)

NOTE: All acquisition parameters must be identical for LSPSIMXX.M and DFTPP2. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (Each instrument is given a unique identifier). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (Each instrument is given a unique identifier).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:

Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Criteria**
51	30.0-60.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

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

GC/MS Operating Conditions - DFTPP	
Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	275°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at @ 1.0 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Organic Department Manager, or senior chemist within the GC/MS group.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

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

The DFTPP tuning standard should also be used to assess the column performance and injection port inertness. Calculate the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, with no evidence of peak tailing. For clients requiring DOD criteria, the tailing factors for these two compounds should not exceed 2.

In order to document the performance of benzidine, pentachlorophenol and DDT, the following procedure must be followed. At the PC, which operates the instrument, load the method TUNETAIL.M into the ENVDA screen. Go into the quant drop down menu and select *calculate/generate report*. When that finishes, select *Qedit quant result.* Each compound can now be evaluated. Double click on benzidine and select *ChromEval* and then *Evaluate tailing*. Follow the instructions given on the screen to evaluate tailing. Send the report to the printer. Repeat the procedure for pentachlorophenol. Repeat the procedure for DDT, selecting *Evaluate degradation*. Follow the instructions given on the screen and then send the report to the printer. The report should be filed with the tune raw data.

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

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270-SIM

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated at six different concentrations, typically, 0.20, 0.50, 1.0, 3.0, 7.0 and 10.0 ng/uL. This is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Some SIM compounds need to be calibrated at higher concentrations. A second standard (Mix B) is prepared containing these compounds. The two standards are combined as in the example below. The full aliquot is used and spiked with the appropriate amount of IS.

Example –

Calibration Mix A is prepared containing ALL analytes at 20 ng/ul.
Calibration Mix B is prepared containing only phenols and phthalates at 20 ng/ul.
For the low standard, 10 ul of mix A and 40 ul of mix B are combined and diluted to 1000 uL with MeCL2. Internal standards are then added prior to analysis.

Cal-Mix A (All Analytes) Added (uL)	Cal-Mix B (Phenols and Phthalates) Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)	Final Conc. Everything but Phenols and Phthalates (ng/uL)	Final Conc. Phenols and phthalates (ng/ul)
10	40	950	1000	0.20	1.0
25	75	900	1000	0.50	2.0
100	100	800	1000	2.0	4.0
70	NA	130	200	7.0	7.0
100	NA	100	200	10	10
150	NA	50	200	15	15

Note: Calibration Mix B only is used to boost the phenols and phthalates concentrations in Cal. levels 1 through 3.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

Column Temperature Program	40°C hold 0.5 minutes 20°/min. to 260°C, hold 0.0 minutes 5°/min to 280°C, hold 0.0 minutes 18°/min to 300°C, hold 4.39 minutes
Final Column Temperature hold	300°C
Run Time	21 minutes
Scan Start Time	2.5 minutes (must be adjusted as column is clipped)
Injection volume	1 uL

The conditions are set up in the method file LSPSIMXX.M

After analysis of the six calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds. This is also applicable to clients that request DOD criteria.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

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

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. Target software calculates the correlation coefficient squared (r^2). This must be equal to or greater than 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.990.

Acceptance criteria independent of calibration model

Either of the two procedures described below may be used to determine calibration function acceptability for linear and non-linear curves. Both % Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

$$\%\,Error = \frac{xi - x'i}{xi}\ x\ 100$$

where:

x'i = Measured amount of analyte at calibration level i, in mass or concentration units
xi= True amount of analyte at calibration level i, in mass or concentration units.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (RSE - expressed as %)

$$RSE = 100\ X \sqrt{\sum_{i=1}^{n} \left| \frac{(x'i - xi)^2}{xi} \right| / (n\text{-}p)}$$

where:

xi= True amount of analyte in calibration level i, in mass or concentration units
x´i= Measured amount of analyte in calibration level i, in mass or concentration units
p= Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)
n= Number of calibration points.

The RSE acceptance limit criterion for the calibration model is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270-SIM. The SSTD1.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. For clients requiring DOD criteria, all project analytes must be within +/- 20% of true value.

7.5.3 Continuing Calibration

A check of the calibration curve must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 1.0 ng/uL.

After quantitation of the 1.0 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin. For clients requiring DOD criteria, all project analytes and surrogates must be within +/- 20%.

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 1.0 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor. Record any of these actions in the appropriate instrument maintenance logbook.
- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may precede using initial calibration response factors.

7.5.4. Retention Time Windows

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

For projects or clients requiring DoD QSM 4.1, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the "Edit entire method" option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap. This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

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

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

7.7.2 Chromatography

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

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

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

For specific manual integration procedures, refer to Katahdin SOP QA-812, “Manual Integration”, current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

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

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.

- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.4 Reporting

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

7.8 Injection Port Liner Cleaning And Silanizing Procedure

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- Take out the liner and rinse it thoroughly with toluene.
- Rinse the liner thoroughly with purge and trap grade methanol.
- Bake the liner in the muffle oven for a minimum of three hours.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

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

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be

extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

Results of the method blank should be less than the PQL/LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. DoD ELAP requires no analyte(s) detected above ½ LOQ (greater than LOQ for common lab contaminants) or greater 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.
If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

The five surrogates (2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10, Pyrene-d10 and 1,4-Dioxane-d8) must meet the current statistically derived or nominal acceptance limits. If statistical limits have not been established then the surrogate recovery must meet the nominal limits of 30-150%. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Non-Conformance Report (NCR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.

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

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

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

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits or nominal limits with the following sporadic exceedance allowances, for DoD clients.

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

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

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

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

For non-DoD clients corrective action is only taken if greater than 10% of the analytes of interest are outside of the laboratory established acceptance limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

All MS/MSD samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

A Corrective Action Report (CAR) must be filled out and filed if any criteria for percent recovery or relative percent difference are not met to document any decisions with reporting data.

9.0 METHOD PERFORMANCE

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

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for

which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

Practical Quantitation Limit (PQL), Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ): These all refer to the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. It is set at the lowest point in the calibration curve for all analyses utilizing an initial calibration.

Limit of Quantitation (LOQ) and Lower Limit of Quantitation (LLOQ) Verifications:

NELAC requires the LOQ be verified annually for each quality system matrix, technology, and analyte. The validity of the LOQ shall be verified by successful analysis of a QC sample containing the analytes of concern in each quality system matrix at 1 to 2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the laboratory established method acceptance criteria or client data quality objectives for accuracy.

In addition to the NELAC requirement, DoD/DOE, requires, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

SW846 requires the laboratory to verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5 to 2 times the established LLOQ.

Therefore, Katahdin will verify the LOQ/LLOQ quarterly for all analyses that are listed on our DoD ELAP Scope of Accreditaion For all other tests, the LOQ/LLOQ verification will be done annually.

The verification acceptance limits are based on our in house statistically derived laboratory control spike limits using ±5 times the standard deviation from the mean (of the LCSs).

Please refer to Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision for additional information.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Method 8270C and Method 8270D.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

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

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

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

Katahdin SOP QA-808, “Generation and Implementation of Statistical QC Limits and/or Control Charts,” current revision.

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. % Error must be ≤ 30%. Refer to section 7.5.2.1 for more details.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. . See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL and/or LOD/LOQ Verification study	Refer to KAS SOP QA-806, “Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications”, current revision.		

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	Correct problem then repeat breakdown check.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	At the beginning of each 12-hour period, prior to analysis of samples.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs ≥ 0.050. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression– coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

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

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2

DoD QSM 4.2 QC REQUIREMENTS

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

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 3

DoD QSM 5.0/5.1 QC REQUIREMENTS

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

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 3

DoD QSM 5.0/5.1 QC REQUIREMENTS

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

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 3

DoD QSM 5.0/5.1 QC REQUIREMENTS

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

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 4

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-14	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 5

ANALYTE QUANITIATION AND INTERNAL STANDARDS

Internal Standard: 1,4-dichlorobenzene-d4
Target and Surrogates:
1,4-Dioxane
1,4-Dioxane-d8 (surrogate)
Benzaldehyde
Phenol
bis(2-Chloroethyl)ether
2-Chlorophenol
2-Methylphenol
3&4-Methylphenol
2,2'-Oxybis(1-chloropropane)
Nitrobenzene
Hexachloroethane
Acetophenone
N-nitroso-di-n-propylamine
1,3-dichlorobenzene
1,4-dichlorobenzene
1,2-dichlorbenzene
Internal Standard: Naphthalene-d8
Target and Surrogates:
Naphthalene
1-Methylnaphthalene (dredge)
2-Methylnaphthalene
2-Methylnaphthalene-D10 (surrogate)
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
bis(2-Chloroethoxy)methane
2,4-Dichlorophenol
4-Chloroaniline
Hexachlorobutadiene
Caprolactam
4-Chloro-3-methylphenol
1,2,4-trichlorobenzene
1,2,4,5-tetrachlorobenzene
Internal Standard: Acenaphthene-d10
Target and Surrogates:
1,1'-Biphenyl (dredge)
2,6 Dimethylnapthalene (dredge)
Acenaphthylene
Acenaphthene
Fluorene
2-Fluorene-d10 (surrogate)
2,4-Dibromophenol (surrogate)
2-Chloronaphthalene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
Dimethylphthalate
2,6-Dinitrotoluene
2,4-Dinitrotoluene
2,4-Dinitrophenol
2,3,4,6-Tetrachlorophenol
Diethylphthalate
4-Chlorophenyl-phenyl ether
4,6-Dinitro-2-methylphenol
N-nitrosodiphenylamine
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
Dibenzofuran
4-Nitrophenol
Internal Standard: Phenanthrene-d10
Target and Surrogates:
Pentachlorophenol
1-Methylphenanthrene (dredge)
Phenanthrene
Hexachlorobenzene (special)
Anthracene
Fluoranthene
Carbazole
Di-n-butylphthalate
4-Bromophenyl-phenyl ether
Atrazine
Internal Standard: Chrysene-d12
Target and Surrogates:
Butylbenzylphthalate
3,3'-Dichlorobenzidine
Pyrene
Benzo(a)Anthracene
Chrysene
Bis-(2-ethylhexyl)phthalate
Pyrene-d10 (surrogate)
Internal Standard: Perylene-d12
Target and Surrogates:
Perylene (dredge)
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(e)pyrene (dredge)
Di-n-octylphthalate
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(ghi)perylene

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 6

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 7

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
1,4-Dioxane-d8	96	66
1,4-Dioxane	88	58
Benzaldehyde	77	105,106
Phenol	94	65,66
bis(2-Chloroethyl)ether	93	63,95
1,3-dichlorobenzene	146	148, 111
1,4-dichlorobenzene	146	148, 111
1,2-dichlorobenzene	146	148, 111
2-Chlorophenol	128	64,130
1,4-Dichlorobenzene-d4 (IS)	152	150,115
2,2'-Oxybis(1-choropropane)	45	77,121
2-Methylphenol	108	107,77
Acetophenone	105	77,51
N-nitroso-di-n-propylamine	70	52,101
Hexachloroethane	117	201,199
3&4-Methylphenol	108	107,77
Nitrobenzene	77	123,51
Isophorone	82	54,138
2-Nitrophenol	139	109,81
1,2,4-trichlorobenzene	180	182, 145
1,2,4,5-tetrachlorobenzene	216	214, 179
2,4-Dimethylphenol	107	122,121
bis(2-Chloroethoxy)methane	93	63,123
2,4-Dichlorophenol	162	164,98
Naphthalene-d8 (IS)	136	137,134
Naphthalene	128	129,127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56
4-Chloro-3-methylphenol	107	77,142
2,4-Dibromophenol (surr)	252	63,143
2-Methylnaphthalene-d10 (surr)	152	150
2-Methylnaphthalene	142	141,115
1-Methylnaphthalene	142	141,115
Hexachlorocyclopentadiene	237	235,239
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
2-Chloronaphthalene	162	127,164
1,1'-Biphenyl	154	153,76
2-Nitroaniline	65	92,138
Dimethylphthalate	163	194,164
2,6-Dinitrotoluene	165	63,89
Acenaphthylene	152	151,153
Acenaphthene	152	154,152
Acenaphthene-d10 (IS)	164	162

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 7

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
3-Nitroaniline	138	65,92
2,4-Dinitrophenol	184	107
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63
4-Nitrophenol	109	139,65
2,3,4,6-Ttrachlorophenol	232	230
Diethylphthalate	149	177,176
Fluorene-d10 (surr)	176	174,178
Fluorene	166	165
4-Chlorophenyl-phenyl ether	204	206,141
4-Nitroaniline	138	108,65
4,6-Dinitro-2-methylphenol	198	121
N-nitrosodiphenylamine	169	168,167
4-Bromophenyl-phenyl ether	248	250,141
Hexachlorobenzene	284	142,249
Atrazine	200	173,215
Pentachlorophenol	266	264,268
Phenanthrene-d10 (IS)	188	189
Phenanthrene	178	179,176
Anthracene	178	179,176
Carbazole	167	166,139
Di-n-butylphthalate	149	150,104
Fluoranthene	202	200,203
Pyrene	202	200,201
Pyrene-d10 (surr)	212	210,106
Butylbenzylphthalate	149	91,206
Benzo(a)anthracene	228	229,226
Chrysene-d12 (IS)	240	236,120
3,3-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
bis(2-Ethylhexyl)phthalate	149	167
Di-n-octylphthalate	149	150
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Perylene-d12 (IS)	264	260
Indeno(1,2,3-cd)pyrene	276	277
Dibenzo(a,h)anthracene	278	279
Benzo(g,h,i)perylene	276	277

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.
(2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

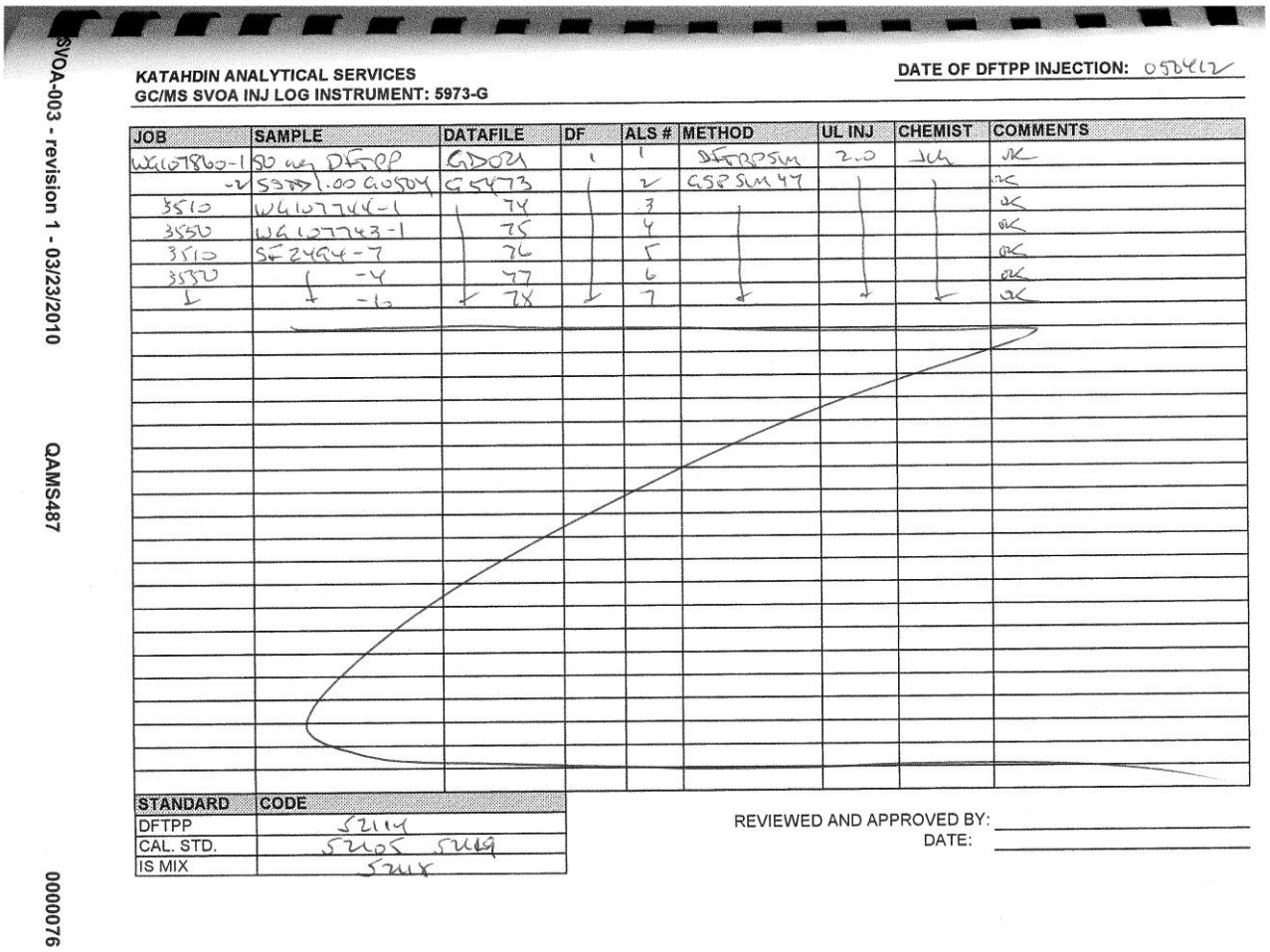
TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE



SVOA-003 - revision 1 - 03/23/2010

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5973-G

DATE OF DFTPP INJECTION: 052412

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
WG107860-1	50 ng DFTPP	GD021	1	1	DFTPPSIM	2.0	JM	OK
-2	5ng/1.00 GC/MS	GC5473		2	GSP SIM 47			OK
3510	WG107744-1	74		3				OK
3550	WG107743-1	75		4				OK
3510	SF 2494-7	76		5				OK
3550	" -4	77		6				OK
"	" -6	78		7				OK

STANDARD	CODE
DFTPP	52114
CAL. STD.	52105 52109
IS MIX	52117

REVIEWED AND APPROVED BY: _____
DATE: _____

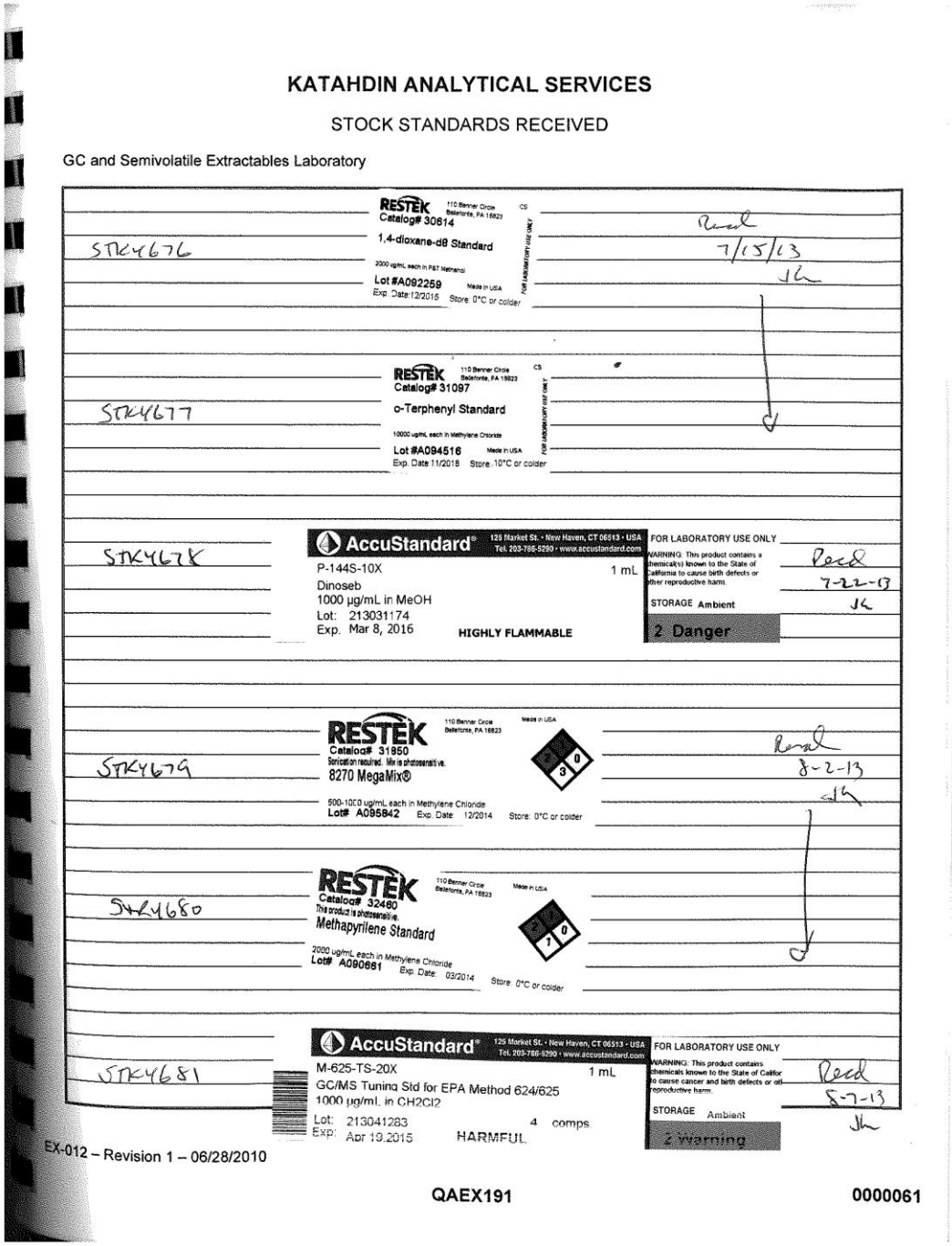
QAMS487

0000076

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY



KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GC and Semivolatile Extractables Laboratory

STK4676 — RESTEK Catalog# 30614, 1,4-dioxane-d8 Standard, 2000 ug/mL each in P&T Methanol, Lot #A092259, Exp. Date 12/2015, Store: 0°C or colder, FOR LABORATORY USE ONLY — Recd 7/15/13 JK

STK4677 — RESTEK Catalog# 31097, o-Terphenyl Standard, 10000 ug/mL each in Methylene Chloride, Lot #A094516, Exp. Date 11/2018, Store: 10°C or colder, FOR LABORATORY USE ONLY

STK4678 — AccuStandard, 125 Market St. • New Haven, CT 06513 • USA, Tel. 203-786-5290 • www.accustandard.com, P-144S-10X, Dinoseb, 1000 µg/mL in MeOH, Lot: 213031174, Exp. Mar 8, 2016, 1 mL, HIGHLY FLAMMABLE, FOR LABORATORY USE ONLY, WARNING: This product contains a chemical(s) known to the State of California to cause birth defects or other reproductive harm., STORAGE Ambient, 2 Danger — Recd 7-22-13 JK

STK4679 — RESTEK Catalog# 31850, Sonication required. Mix is photosensitive., 8270 MegaMix®, 500-1000 ug/mL each in Methylene Chloride, Lot# A095842, Exp. Date 12/2014, Store: 0°C or colder, Made in USA — Recd 8-2-13 JK

STK4680 — RESTEK Catalog# 32460, This product is photosensitive., Methapyrilene Standard, 2000 ug/mL each in Methylene Chloride, Lot# A090881, Exp. Date: 03/2014, Store: 0°C or colder, Made in USA

STK4681 — AccuStandard, 125 Market St. • New Haven, CT 06513 • USA, Tel. 203-786-5290 • www.accustandard.com, M-625-TS-20X, GC/MS Tuning Std for EPA Method 624/625, 1000 µg/mL in CH2Cl2, Lot: 213041283, Exp: Apr 19,2015, 1 mL, 4 comps, HARMFUL, FOR LABORATORY USE ONLY, WARNING: This product contains chemicals known to the State of California to cause cancer and birth defects or other reproductive harm., STORAGE Ambient, 2 Warning — Recd 5-7-13 JK

EX-012 – Revision 1 – 06/28/2010

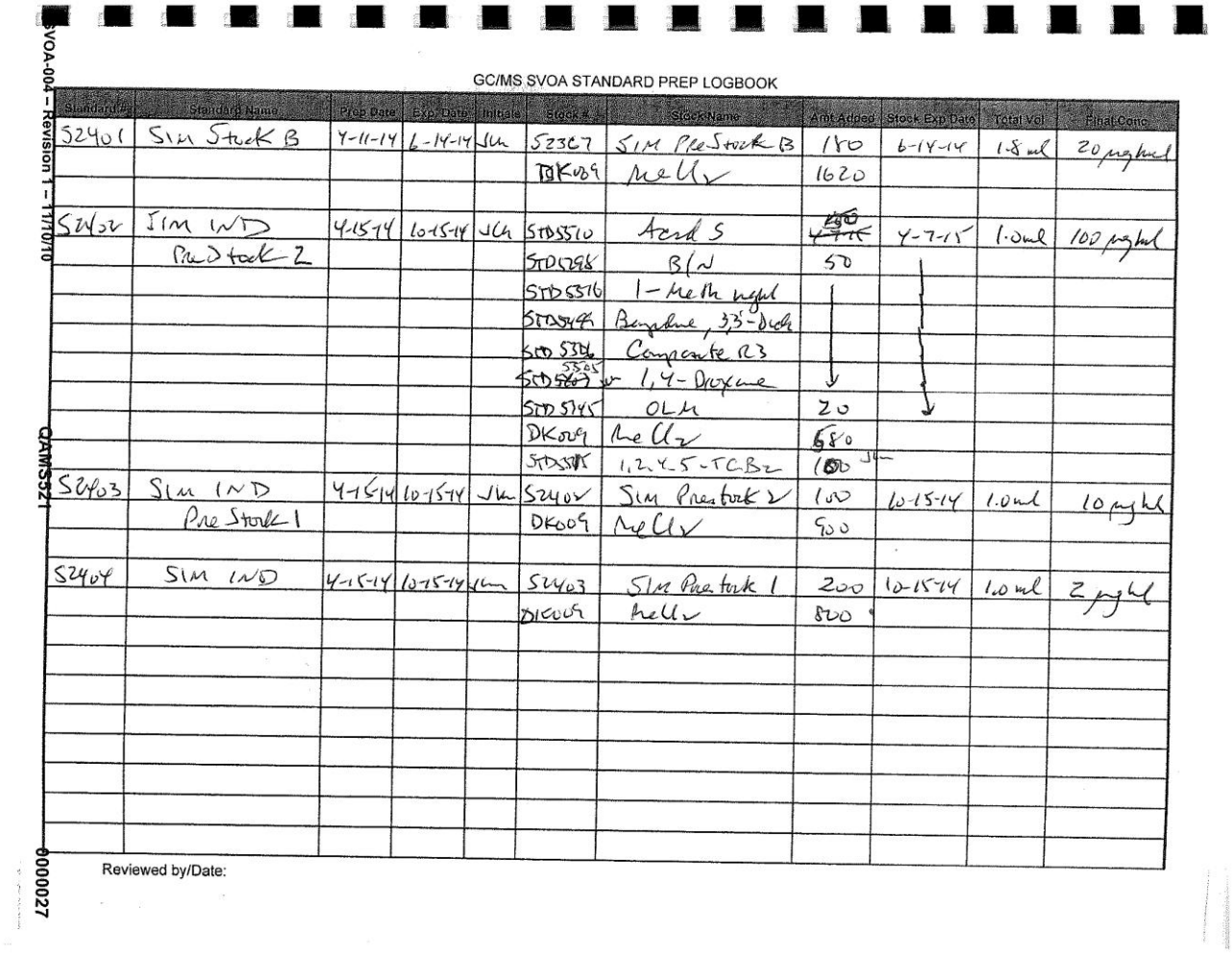
QAEX191

0000061

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY



SVOA-004 – Revision 1 – 11/10/10

GC/MS SVOA STANDARD PREP LOGBOOK

Standard ID	Standard Name	Prep Date	Exp Date	Initials	Stock #	Stock Name	Amt Added	Stock Exp Date	Total Vol	Final Conc
S2401	SIM Stock B	4-11-14	6-14-14	JLH	52367	SIM PreStock B	180	6-14-14	1.8 ml	20 µg/ml
					DK009	MeCl2	1620			
S2402	SIM IND PreStock 2	4-15-14	10-15-14	JLH	STD5510	Acid S	~~450~~ 440	4-7-15	1.0ml	100 µg/ml
					STD5298	B/N	50			
					STD5516	1-Meth napht				
					STD5498	Benzidine, 3,3'-Dich				
					STD5336	Composite R3				
					STD5503 3304	1,4-Dioxane				
					STD5345	OLM	20			
					DK009	MeCl2	680			
					STD5317	1,2,4-5-TCB2	100 JLH			
S2403	SIM IND PreStock 1	4-15-14	10-15-14	JLH	S2402	SIM Prestock 2	100	10-15-14	1.0ml	10 µg/ml
					DK009	MeCl2	900			
S2404	SIM IND	4-15-14	10-15-14	JLH	S2403	SIM Prestock 1	200	10-15-14	1.0 ml	2 µg/ml
					DK009	MeCl2	800			

Reviewed by/Date:

CAMS5271

0000027

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

ADDENDUM 1

LOW LEVEL 1,4-DIOXANE ANALYSIS

The following are differences from the standard 8270 C or D SIM analysis:

GC Operating Conditions – The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP (Sect. 7.4) with the following exceptions:

Column Temperature Program	35°C hold 3 minutes 20°/min. to 300°C
Final Column Temperature hold	300°C
Run Time	16.25 minutes
Scan Start Time	2.3 minutes (must be adjusted as column is clipped)
Injection volume	1 uL

Stock Standards – 1,4-Dioxane and 1,4-Dioxane each at 20 ug/mL

Calibration Standards – Use the above stock standards to prepare calibration standards at concentrations 0.25 ug/mL, 0.50 ug/mL, 1.0 ug/mL, 2.0 ug/mL, 4.0 ug/mL and 6.0 ug/mL. The 1.0 ug/mL is also the continuing calibration verification standard.

Sample analysis – Add 1 uL of internal standard (Section 5.3.2.4) aliquot of sample.

The ions for 1,4-Dioxane are 58 and 88.
The ions for 1,4-Dioxane-d8 are 64 and 96.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Micheal Thomas Date: 07-24-00

Approved By:

Department Manager: [signature] Date: 6-23-06

Operations Manager: Deborah J. Nadeau Date: 6.23.06

QA Officer: Susie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03	Changes to Sect. 5.5 & Figures 3 & 4 to reflect current spike solutions and concentrations Replaced cover page. Original cover page filed with SOP CA502-02	LAD	04/06	04/06
04	Added definitions, added waste information added LCSD, added SIM LCS/D, MS/D, updated Table 1, added use of narrow range pH paper. Minor changes throughout to reflect current practice.	LAD	09/07	09/07
05	Removed MS/MSD 14 day requirement. Changed CLLE extraction time to 18 → 24 hours. Added information on determining initial sample volume. Added extracted sample disposal. Removed all references to method 625.	LAD	09/08	09/08
06	Added to check pH after B/N CLLE extraction to ensure pH ≥ 11. If not add more NaOH and continue extracting. Added information for initial volume determination. Added reference to CA-109. Updated logbook example. Added if extract goes dry - re extract.	LAD	10/09	10/09
07	Sect. 5 - Removed baking and rinsing Na_2SO_4. Added 1,4-Dioxane to SIM surrogate Mix. Sect. 7 added acid to B/N SIM., removed to let separate for 10 minutes, minor edits throughout.	LAD	03/12	03/12

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08	Removed Sect. 7.1.9, determining the sample initial volume. Sect. 7.1.4 has this information. Figures 1 and 2 updated.	LAD	05/13	05/13
09	Sect. 5 - Updated prep of SIM/scan Surrogate mix. Sect. 7 - Updated Surrogate Spiking directions. Updated Figure 1.	LAD	06/14	06/14
10	Sect. 7 - For separatory funnel, corrected extraction sequence (acid then basic), and that the pH is determined after first shake. Updated Solvent Lot Check Form. Changed KAS INC → KAS	LAD	08/15	08/15
11	Change order of pH checking and spike stds additions. Replace use of HCl with H_2SO_4 to adjust pH. Added test for residual chlorine. Updated method references for NELAC, DOD + SW 846.	LAD	09/17	09/17

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

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

I acknowledge receipt of copy ___ of document **SOP CA-502-11**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-502-11**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____ Date: _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe procedures utilized by Katahdin Analytical personnel in the preparation of all non-CLP aqueous samples for analysis of extractable semivolatile organic compounds.

The goal of this procedure is to ensure uniformity involving the preparation of samples for subsequent SVOA analysis by GC/MS. This SOP is applicable to EPA Methods 3510 (modified separatory funnel extraction) and 3520 (continuous liquid-liquid extraction), current revisions.

1.1 Definitions

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

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

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

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

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

1.3 Safety

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

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

1.4 Pollution Prevention/Waste Disposal

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

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware, disassembly of CLLEs after extraction, etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction aqueous samples are considered either N-Hi or N-Low waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Sodium sulfate used for sample drying should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

For aqueous samples extracted by CLLE, a one liter aliquot of sample is adjusted to pH $\leq$ 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. The pH is then adjusted to pH $\geq$ 11 and the sample is extracted again with methylene chloride. A modified separatory funnel extraction may also be used. If this procedure is used, the sample aliquot is first adjusted to pH $\geq$ 11 and then to pH $\leq$ 2. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC/MS analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure that clean glassware and apparatus are used and pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Brand names and catalog numbers are included for illustration purposes only.

4.1 Continuous liquid-liquid extractors - including body, 500 mL round bottom flask and Alhin condensers and equipped with Teflon or glass connecting joints requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent).

4.2 Glass powder funnels.

4.3 Fluted filter paper, 18.5cm diameter.

4.4 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

4.5 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with neck clips.

4.6 Snyder column - Kuderna-Danish, three- or four-ball macro (Kontes K-503000-0121 or equivalent).

4.7 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.8 Vials - Glass, 1.8 mL capacity, with polytetrafluoroethylene (PTFE)-lined screw top and 12 mL with Teflon-lined caps.

4.9 2 L separatory funnel, equipped with Teflon stopper and stopcock; Nalgene Teflon FEP separatory funnels may also be used.

4.10 Organic Free Boiling Chips - approximately 10/40 mesh, Teflon or silicon carbide (or equivalent). Cleaned by Soxhlet for 18 hours.

4.11 Water bath - heated, with concentric ring cover, capable of temperature control (± 20°C). The bath should be used in a hood.

4.12 Nitrogen evaporation apparatus.

4.13 Wide range pH test strips, pH 0-14, Whatman CF Type.

4.14 Glass rods for stirring samples.

4.15 Amber bottles or other appropriate containers for collection of extracts from separatory funnel extraction.

4.16 5 ¾" Pasteur pipets.

4.17 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.18 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS AND STANDARDS

All reagent and solvent lots must be checked for possible contamination. Refer to the current version of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details. The extraction staff is responsible for submitting samples to the GC or GC/MS sections for appropriate analysis. All information concerning preparation of the reagent/solvent lot sample will be recorded in the Organic Extraction Log (Figure 1) and acceptance or rejection of these lots must be recorded in the solvent/reagent lot check logbook (Figure 2). All reagents and solvents must be free (<PQL) of any target compounds.

5.1 Laboratory Reagent Grade Water - defined as water in which an interferent is not observed at or above the PQL of each parameter of interest. Deionized water filtered through activated charcoal.

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, certified by the manufacturer/vendor as purified.

5.3 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) – Prepared in an icebath by slowly adding a volume of concentrated H_2SO_4 to an equivalent volume of reagent water and swirl gently to mix. Caution should be taken when adding the acid to the water as the reaction is highly exothermic.

5.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent, evaluated prior to use by concentration of 200 mLs to 1.0 mL followed by GC and/or GC/MS analysis.

5.5 Standard Preparation - For all standard preparations, see current revision of the following Katahdin Analytical SOPs:

- "Standards Preparation, Documentation and Traceability", (CA-106, current revision)
- "Balance Calibration," (CA-102, current revision)

5.5.1. SCAN/SIM Surrogate Spiking Solution – A solution containing surrogate spike for both semivolatile SCAN and SIM analysis - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound - SCAN	**Conc.**
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL
Compound - SIM	**Conc.**
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	20 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.2 SVOA Matrix Spike/Lab Control Samples Spiking Solution - the matrix spike/LCS solution consists of the compounds listed in Figure 3.
Prepare a spiking solution that contains each of the base/neutral compounds listed in Figure 3 at 50 ug/mL in methanol and the acid compounds at 100 ug/mL in methanol. Matrix spike/LCS standards are stored in the freezer (-10°C to -20°C) located in the storage area.

5.5.3 Base/Neutral/Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 2 ug/mL for base/neutrals and 4.0 ug/mL for acids. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months or sooner if comparison with quality control check samples indicates a problem.

5.5.4 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 4 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These

solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.5.5 Potassium iodide starch paper

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Continuous liquid-liquid (Method 3520) and/or separatory funnel (Method 3510) extractions for semivolatiles must be started within seven days of date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific. If sampling date is unknown, the hold time is counted from one day prior to date received.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- CLLE start time and end time
- Lot number of the vials the concentrated extracts are stored in.

Follow the proper procedures for maintaining Internal Chain of Custodies for samples when removing and replacing samples in storage locations. This procedure is described in KAS SOP SD-902, “Sample Receipt and Internal Control”, current revision.

7.1 CONTINUOUS LIQUID-LIQUID EXTRACTION (Method 3520)

7.1.1 Set up the CLLE apparatus. All glassware should be pre-rinsed three times with methylene chloride in order to eliminate any contamination factors.

7.1.2 Add approximately 500 - 600 mL of methylene chloride to the CLLE body. Label each flask with the following: sample number (or QC identification number), analyte (SVOA), extraction method (CLLE), and extraction date.

7.1.3 A method blank and a laboratory control sample (LCS) must be prepared for each daily extraction batch of twenty samples or fewer (if a work order consists of more than twenty samples, a new batch must be started on a separate page with its own method blank and LCS). To prepare method blank and LCS, add 1 L reagent water to a CLLE body. Be sure that no water leaks into the round bottom flask. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.1.4 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.1.5 Transfer the sample to a CLLE body slowly, being sure that no water leaks into the round bottom flask.

7.1.6 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to CLLE bodies for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable a laboratory control sample duplicate (LCSD) may be substituted.

7.1.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL SCAN/SIM surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use. (sect. 5.5.2). **NOTE**: If REQUEST is for both SCAN and SIM, an LCS/LCSD and/or MS/MSD are required for each analysis.

7.1.8 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.1.10.1 If the request is for SVOA - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3).

7.1.10.2 If the request is for SIM - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and add 1.0 mL of Base/Neutral/Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.4).

7.1.10.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution -add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and add 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.5).

7.1.9 Check the pH of each sample with wide range pH paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod. Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to $\leq$ pH 2 with 1:1 H_2SO_4 after addition of surrogates and spikes and prior to attaching Allihn condensers (Step 7.1.11). Stir with a glass stirring rod and check pH by tapping the glassrod onto wide range pH paper. The pH must be ≤ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used. Also test samples for residual chlorine with potassium iodide starch paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod.

7.1.10 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.1.11 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for 18 to 24 hours. Turn off the mantles and let samples cool.

7.1.12 Detach condensers and verify that the pH is still $\leq$ 2 in the same manner mentioned in 7.1.6. If the pH has changed, more acid should be added to make the pH $\leq$ 2 and the sample extracted for several more hours.

7.1.13 Upon completion of acid extraction, allow the sample to cool. Detach condensers and add enough 10N NaOH to adjust the pH to $\geq$ 11 with stirring. Use glass stirring rods to stir and check the pH of each sample in the same manner mentioned in 7.1.6.

7.1.14 Re-attach Allihn condensers, turn on heating mantles, and allow samples to extract for 18 to 24 hours. Turn off mantles and allow samples to cool.

7.1.15 Detach condensers and verify that the pH is still $\geq$ 11 in the same manner mentioned in 7.1.6. If the pH has changed, more NaOH should be added to make the pH $\geq$ 11 and the sample extracted for several more hours.

7.1.16 Once samples are cool to the touch, the CLLE apparatus can be disassembled. The round bottom flask is removed, covered with foil and placed in the interim extract refrigerator. The remaining sample in the CLLE body is poured in the "N-Hi" satellite.

7.1.17 Proceed to Step 7.3 for sample extract concentration procedures.

7.2 SEPARATORY FUNNEL EXTRACTION (Modified Method 3510)

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.2.1 Rinse all glassware, including teflon separatory funnels, three times with methylene chloride prior to use.

7.2.2 Label 2 L separatory funnels and amber collection bottles clearly. Each label should include: sample number (or QC indicator number), analyte (SVOA), matrix (Aq), extraction date.

7.2.3 A method blank and a laboratory control sample (LCS) must be prepared for every 20 samples or with each extraction batch, whichever is more frequent. To prepare method blank and LCS, add 1 L reagent water to a separatory funnel. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.2.4 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.2.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to separatory funnels for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable, a laboratory control sample duplicate (LCSD) may be substituted.

7.2.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL SCAN/SIM surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use. (sect. 5.5.2). **NOTE**: If REQUEST is for both SCAN and SIM, an LCS/LCSD and/or MS/MSD are required for each analysis.

7.2.7 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in the extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.2.7.1 If the request is for SVOA, use the SVOA Spiking Solution.

7.2.7.2 If the request is for SIM, use the SIM Spiking solution.

7.2.7.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution

7.2.8 For each sample, rinse the original sample container with 60 mL of methylene chloride. Add this rinse to the separatory funnel.

7.2.9 Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to pH ≤ 2 with 1:1 H_2SO_4 after addition of surrogates and spikes. Also test samples for residual chlorine with potassium iodide starch paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod.

7.2.10 Add 60 mL of methylene chloride directly to the method blank and LCS/LCSD separatory funnels.

7.2.11 Extract the samples by shaking the funnel for two minutes, venting often, but gently, in a hood to release pressure. A mechanical shaker may be used, where samples are shaken for 3 minutes.

7.2.12 After the first shake dip a glass stirring rod into the sample and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be ≤ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used.

7.2.13 Allow phases to separate. Drain the methylene chloride layer into an amber collection bottle.

7.2.14 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation. Such means include swirling, centrifugation, and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor (CLLE) may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook, and the batch transferred to a CLLE batch with its own batch ID.

7.2.15 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time. Collect the methylene chloride layer in the same amber collection bottle.

7.2.16 Repeat the extraction for a third time. Collect the methylene chloride layer in the same amber collection bottle.

7.2.17 Following the third shake, adjust the pH to $\geq$ 11 with 10N NaOH. Add enough 10N NaOH to adjust the pH to $\geq$ 11.

7.2.18 Add 60 mL methylene chloride to each separatory funnel and extract the samples in the same manner described in 7.2.11 – 7.2.14. Collect the methylene chloride layer in the same amber collection bottle used to collect the acid fraction.

7.2.19 After the first shake dip a glass stirring rod into the sample and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be ≥ 11. If the pH test strip does not clearly indicate the pH is greater than 11, narrow range pH paper must be used.

7.2.20 Repeat 2 mores times with 2 more 60 mL aliquots of methylene chloride Collect the methylene chloride layer in the same amber collection bottle

7.2.21 Sample waste should be poured into the “N-Hi” satellite.

7.2.22 Proceed to Section 7.3 for extract concentration procedures.

7.3 CONCENTRATING THE EXTRACTS

7.3.1 For Methods 3510 and 3520, the combined fractions are concentrated to a final volume of 1.0 mL.

7.3.2 Rinse the K-D glassware (flask, concentration tube, and snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride. Add two boiling chips to the K-D prior to final rinse. Also rinse the assembled funnels, filter paper, and granular sodium sulfate

used for drying the extracts. Record the lot numbers for filter paper, sodium sulfate crystals and methylene chloride in the extractions logbook.

7.3.3 Transfer the methylene chloride extract to a K-D concentrator setup through a short stem funnel filled with 1-2 inches of sodium sulfate in filter paper. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain

7.3.4 Transfer the label from the collection bottle or round bottom flask (for CLLE) to a K-D. Remove the funnel and attach a 3- or 4-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.3.5 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.3.6 Reduce the methylene chloride extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.3.7 Reduce each extract to slightly less than 1 mL and then, using a 5 ¾" pasteur pipet, transfer the final extract and label to a 1.8 mL vial with PTFE-lined cap.

7.3.8 If at any time during the concentration process the concentrator tube goes dry, reextraction must occur immediately.

7.3.9 Transfer all of the extract to a 1.8 mL screw cap vial. Using methylene chloride, adjust the final volume of each extract to 1 mL by comparison to an appropriate reference vial.

Store in refrigerator until GC/MS analysis.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:
- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:
- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of semivolatiles for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015), Methods 3510 and 3520, current revisions.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 5.1, January 2017.

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

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

LIST OF TABLES AND FIGURES

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-11	METHOD 3510, current revision
Apparatus/Materials	1) 250 mL amber bottle or flask 2) 1.0 mL syringe 3) short stem funnels	1) 250 mL Erlenmeyer flask 2) 5.0 mL syringe 3) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract collection in amber bottle or Erlenmeyer flask 2) Add surrogate/spike to sample in CLLE 3) Extract for 3 minutes on mechanical shaker 4) extract three times at pH $\geq$ 11, then extract three times at pH $\leq$ 2. 5) extract dried using Na_2SO_4 in short stem funnels 6) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7) water bath temp 75-85 deg C 8) no apparatus height specification for concentration on water bath 9) sample removed from water bath when volume reaches ~6 mL 10) N bath temp no higher than 39 deg C	1) extract collection in Erlenmeyer flask 2) Add surrogate/spike directly to sample bottle 3) Extract by shaking vigorously for 1 - 2 minutes with periodic venting 4) extract three times at pH $\leq$ 2, then extract three times at pH $\geq$ 11. 5) extract dried using Na_2SO_4 in drying columns 6) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 7) water bath temp 15-20 deg C above solvent boiling temp 8) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 9) sample removed from water bath when volume reaches 1 mL 10) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1, continued

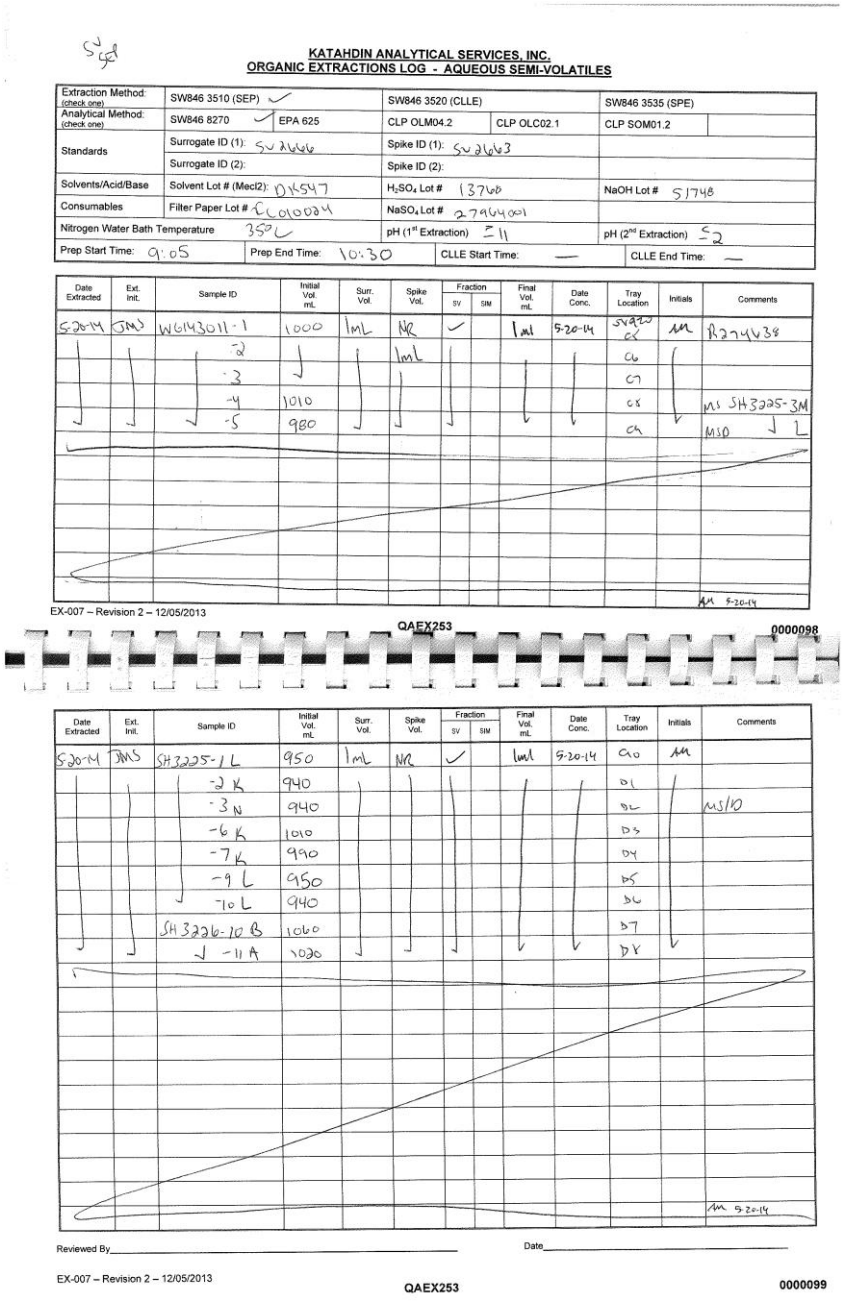
SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-502-11	METHOD 3520, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) Add surrogate/spike to sample in CLLE 2) Add approximately 500 - 600 mL of methylene chloride to the CLLE body 3) CLLE for 22 $\pm$ 2 hours 4) Extract dried using Na_2SO_4 in short stem funnels 5) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 6) water bath temp 75-85 deg C 7) no apparatus height specification for concentration on water bath 8) sample removed from water bath when volume reaches ~6 mL 9) N bath temp no higher than 39 deg C	1) Add surrogate/spike directly to sample bottle 2) Add 300 - 500 mL of methylene chloride to the distilling flask of the extractor 3) CLLE for 18 - 24 hours 4) Extract dried using Na_2SO_4 in drying columns 5) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 6) water bath temp 15-20 deg C above solvent boiling temp 7) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 8) sample removed from water bath when volume reaches 1 mL 9) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 1

EXAMPLE OF SEMIVOLATILES LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS SEMI-VOLATILES

Extraction Method: (check one)	SW846 3510 (SEP) ✓		SW846 3520 (CLLE)		SW846 3535 (SPE)
Analytical Method: (check one)	SW846 8270 ✓	EPA 625	CLP OLM04.2	CLP OLC02.1	CLP SOM01.2
Standards	Surrogate ID (1): SV2646		Spike ID (1): SV2663		
	Surrogate ID (2):		Spike ID (2):		
Solvents/Acid/Base	Solvent Lot # (MeCl2): DK547		H_2SO_4 Lot # 13760		NaOH Lot # 51748
Consumables	Filter Paper Lot # FC010024		$NaSO_4$ Lot # 2796400		
Nitrogen Water Bath Temperature	35°C		pH (1st Extraction) ~11		pH (2nd Extraction) ~2
Prep Start Time: 9:05	Prep End Time: 10:30		CLLE Start Time: —		CLLE End Time: —

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SIM	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
5-20-14	JMS	W0143011-1	1000	1mL	NR	✓		1ml	5-20-14	SV920 C5	AM	R274638
		-2			1mL					C6		
		-3								C7		
		-4	1010							C8		MS SH3225-3M
		-5	980							C9		MSD ✓ L

AM 5-20-14

EX-007 – Revision 2 – 12/05/2013 QAEX253 0000098

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SIM	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
5-20-14	JMS	SH3225-1L	950	1mL	NR	✓		1ml	5-20-14	C10	AM	
		-2K	940							D1		
		-3N	940							D2		MS/D
		-6K	1010							D3		
		-7K	990							D4		
		-9L	950							D5		
		-10L	940							D6		
		SH3226-10B	1060							D7		
		✓ -11A	1020							D8		

AM 5-20-14

Reviewed By_____ Date_____

EX-007 – Revision 2 – 12/05/2013 QAEX253 0000099

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 2

SOLVENT LOT CHECK LOGBOOK

KATAHDIN ANALYTICAL SERVICES

SOLVENT LOT CHECK

SOLVENT:_____

LOT#:_____

DATE RECEIVED:_____

DATE CONCENTRATED:_____

CONCENTRATED BY:_____

PREP METHOD:_____

TRAY LOCATION:_____

ANALYZED BY:_____

PASS/FAIL:_____

EX-027 – Revision 1 – 08/15/2015

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 3

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis(2-Ethylhexyl)adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl phthalate
3,3'-Dichlorobenzidine	Diethyl adipate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 4

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP:

Review Date: 02-23-2018

SOP Number: CA-502-11

SOP Title: PREPERATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 2-23-18

QAO Signature: Lucie Dimond **Date:** 02.26.18

CERTIFICATE OF ACCREDITATION

ANSI-ASQ National Accreditation Board
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

TestAmerica Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

has been assessed by ANAB
and meets the requirements of international standard

ISO/IEC 17025:2005

and DoD Quality Systems Manual for Environmental Laboratories (DoD QSM V 5.1)

while demonstrating technical competence in the fields of

TESTING

Refer to the accompanying Scope of Accreditation for information regarding the types of calibrations and/or tests to which this accreditation applies.

L2468
Certificate Number

ANAB Approval

Certificate Valid: 03/22/2018-01/20/2021
Version No. 003 Issued: 03/22/2018

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

ANSI-ASQ National Accreditation Board

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005 AND DOD QUALITY SYSTEMS MAUAL FOR ENVIRONMENTAL LABORATORIES (DOD QSM V5.1)

TestAmerica Sacramento
880 Riverside Parkway
West Sacramento, CA 95605
Ms. Lisa Stafford
916-373-5600

TESTING

Valid to: **January 20, 2021** Certificate Number: **L2468**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Aluminum
ICP-AES	EPA 6010B/6010C	Antimony
ICP-AES	EPA 6010B/6010C	Arsenic
ICP-AES	EPA 6010B/6010C	Barium
ICP-AES	EPA 6010B/6010C	Beryllium
ICP-AES	EPA 6010B/6010C	Boron
ICP-AES	EPA 6010B/6010C	Cadmium
ICP-AES	EPA 6010B/6010C	Calcium
ICP-AES	EPA 6010B/6010C	Chromium (Total)
ICP-AES	EPA 6010B/6010C	Cobalt
ICP-AES	EPA 6010B/6010C	Copper
ICP-AES	EPA 6010B/6010C	Iron
ICP-AES	EPA 6010B/6010C	Lead
ICP-AES	EPA 6010B/6010C	Magnesium
ICP-AES	EPA 6010B/6010C	Manganese
ICP-AES	EPA 6010B/6010C	Molybdenum
ICP-AES	EPA 6010B/6010C	Nickel
ICP-AES	EPA 6010B/6010C	Potassium
ICP-AES	EPA 6010B/6010C	Selenium
ICP-AES	EPA 6010B/6010C	Silica

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Silicon
ICP-AES	EPA 6010B/6010C	Silver
ICP-AES	EPA 6010B/6010C	Sodium
ICP-AES	EPA 6010B/6010C	Thallium
ICP-AES	EPA 6010B/6010C	Tin
ICP-AES	EPA 6010B/6010C	Titanium
ICP-AES	EPA 6010B/6010C	Vanadium
ICP-AES	EPA 6010B/6010C	Zinc
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel
ICP-MS	EPA 6020/6020A	Phosphorus
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium
ICP-MS	EPA 6020/6020A	Strontium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Tin
ICP-MS	EPA 6020/6020A	Titanium
ICP-MS	EPA 6020/6020A	Uranium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc

Non-Potable Water		
Technology	**Method**	**Analyte**
CVAAS	EPA 7470A	Mercury
Colorimetric	EPA 353.2	Nitrate
Colorimetric	EPA 353.2	Nitrate-nitrite
Colorimetric	EPA 353.2	Nitrite
Colorimetric	EPA 410.4	Chemical Oxygen Demand (COD)
LC/MS/MS	EPA 6850	Perchlorate
Colorimetric	EPA 7196A	Chromium (Hexavalent)
Probe	EPA 9040B/9040C	pH
Ion Chromatography	EPA 9056A/300.0	Bromide
Ion Chromatography	EPA 9056A/300.0	Chloride
Ion Chromatography	EPA 9056A/300.0	Fluoride
Ion Chromatography	EPA 9056A/300.0	Nitrate
Ion Chromatography	EPA 9056A/300.0	Nitrite
Ion Chromatography	EPA 9056A/300.0	Orthophosphate
Ion Chromatography	EPA 9056A/300.0	Sulfate
Titration	SM 2320B	Alkalinity
Gravimetric	SM 2540B	Solids, Total
Gravimetric	SM 2540C	Solids, Total Dissolved
Gravimetric	SM 2540D	Solids, Total Suspended
Colorimetric/Hydrolysis	EPA 353.2 Modified / WS-WC-0050	Nitrocellulose
GC/MS	EPA 8260B/8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/8260C	1,2-Dibromoethane
GC/MS	EPA 8260B/8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,2-Dichloroethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,3-Dichloropropane
GC/MS	EPA 8260B/8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1-Chlorohexane
GC/MS	EPA 8260B/8260C	2,2-Dichloropropane
GC/MS	EPA 8260B/8260C	2-Butanone (MEK)
GC/MS	EPA 8260B/8260C	2-Chlorotoluene
GC/MS	EPA 8260B/8260C	2-Hexanone (MBK)
GC/MS	EPA 8260B/8260C	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA 8260B/8260C	4-Chlorotoluene
GC/MS	EPA 8260B/8260C	4-Isopropyltoluene
GC/MS	EPA 8260B/8260C	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 8260B/8260C	Acetone
GC/MS	EPA 8260B/8260C	Allyl Chloride
GC/MS	EPA 8260B/8260C	Benzene
GC/MS	EPA 8260B/8260C	Bromobenzene
GC/MS	EPA 8260B/8260C	Bromochloromethane
GC/MS	EPA 8260B/8260C	Bromodichloromethane
GC/MS	EPA 8260B/8260C	Bromoform
GC/MS	EPA 8260B/8260C	Bromomethane
GC/MS	EPA 8260B/8260C	Carbon Disulfide
GC/MS	EPA 8260B/8260C	Carbon Tetrachloride
GC/MS	EPA 8260B/8260C	Chlorobenzene
GC/MS	EPA 8260B/8260C	Chloroethane
GC/MS	EPA 8260B/8260C	Chloroform
GC/MS	EPA 8260B/8260C	Chloromethane
GC/MS	EPA 8260B/8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Cyclohexane
GC/MS	EPA 8260B/8260C	Dibromochloromethane
GC/MS	EPA 8260B/8260C	Dibromomethane
GC/MS	EPA 8260B/8260C	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C	Diisopropyl Ether (DIPE)
GC/MS	EPA 8260B/8260C	Ethylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	Ethylmethacrylate
GC/MS	EPA 8260B/8260C	Ethyl tert-butyl Ether (ETBE)
GC/MS	EPA 8260B/8260C	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C	Hexane
GC/MS	EPA 8260B/8260C	Iodomethane
GC/MS	EPA 8260B/8260C	Isobutanol (2-Methyl-1-propanol)
GC/MS	EPA 8260B/8260C	Isopropylbenzene
GC/MS	EPA 8260B/8260C	m & p Xylene
GC/MS	EPA 8260B/8260C	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA 8260B/8260C	Methylene Chloride
GC/MS	EPA 8260B/8260C	Naphthalene
GC/MS	EPA 8260B/8260C	n-Butylbenzene
GC/MS	EPA 8260B/8260C	n-Propylbenzene
GC/MS	EPA 8260B/8260C	o-Xylene
GC/MS	EPA 8260B/8260C	sec-Butylbenzene
GC/MS	EPA 8260B/8260C	Styrene
GC/MS	EPA 8260B/8260C	t-Amyl methyl Ether (TAME)
GC/MS	EPA 8260B/8260C	t-1,4-Dichloro-2-Butene
GC/MS	EPA 8260B/8260C	tert-Butylbenzene
GC/MS	EPA 8260B/8260C	Tetrachloroethene
GC/MS	EPA 8260B/8260C	Toluene
GC/MS	EPA 8260B/8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Trichloroethene
GC/MS	EPA 8260B/8260C	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C	Vinyl Acetate
GC/MS	EPA 8260B/8260C	Vinyl Chloride
GC/MS	EPA 8260B/8260C	Xylenes, Total
GC/MS	EPA 8260B/AK101MS	Gasoline (GRO)
GC/MS	EPA 8270C/8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270C/8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270C/8270D	1,4-Dichlorobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	1-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dimethylphenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrophenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2,6-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2-Chloronaphthalene
GC/MS	EPA 8270C/8270D	2-Chlorophenol
GC/MS	EPA 8270C/8270D	2-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2-Methylphenol
GC/MS	EPA 8270C/8270D	2-Nitroaniline
GC/MS	EPA 8270C/8270D	2-Nitrophenol
GC/MS	EPA 8270C/8270D	3&4-Methylphenol
GC/MS	EPA 8270C/8270D	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/8270D	3-Nitroaniline
GC/MS	EPA 8270C/8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/8270D	4-Chloroaniline
GC/MS	EPA 8270C/8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Nitroaniline
GC/MS	EPA 8270C/8270D	4-Nitrophenol
GC/MS	EPA 8270C/8270D	Acenaphthene
GC/MS	EPA 8270C/8270D	Acenaphthylene
GC/MS	EPA 8270C/8270D	Aniline
GC/MS	EPA 8270C/8270D	Anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)pyrene
GC/MS	EPA 8270C/8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/8270D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/8270D	Benzoic Acid

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	Benzyl Alcohol
GC/MS	EPA 8270C/8270D	Benzyl butyl Phthalate
GC/MS	EPA 8270C/8270D	Biphenyl
GC/MS	EPA 8270C/8270D	Bis(2-chloroethoxy) Methane
GC/MS	EPA 8270C/8270D	Bis(2-chloroethyl) Ether
GC/MS	EPA 8270C/8270D	Bis(2-chloroisopropyl) Ether
GC/MS	EPA 8270C/8270D	Carbazole
GC/MS	EPA 8270C/8270D	Chrysene
GC/MS	EPA 8270C/8270D	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA 8270C/8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/8270D	Dibenzofuran
GC/MS	EPA 8270C/8270D	Diethyl Phthalate
GC/MS	EPA 8270C/8270D	Dimethyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-butyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-octyl Phthalate
GC/MS	EPA 8270C/8270D	Fluoranthene
GC/MS	EPA 8270C/8270D	Fluorene
GC/MS	EPA 8270C/8270D	Hexachlorobenzene
GC/MS	EPA 8270C/8270D	Hexachlorobutadiene
GC/MS	EPA 8270C/8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/8270D	Hexachloroethane
GC/MS	EPA 8270C/8270D	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA 8270C/8270D	Isophorone
GC/MS	EPA 8270C/8270D	Naphthalene
GC/MS	EPA 8270C/8270D	Nitrobenzene
GC/MS	EPA 8270C/8270D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/8270D	Pentachlorophenol
GC/MS	EPA 8270C/8270D	Phenanthrene
GC/MS	EPA 8270C/8270D	Phenol
GC/MS	EPA 8270C/8270D	Pyrene
GC/MS	EPA 8270C/8270D	Pyridine
GC/MS SIM	EPA 8260C-SIM	1,1,2-Trichloroethane
GC/MS SIM	EPA 8260C-SIM	1,1,2,2-Tetrachloroethane
GC/MS SIM	EPA 8260C-SIM	1,2,3-Trichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8260C-SIM	1,2-Dibromoethane
GC/MS SIM	EPA 8260C-SIM	1,2-Dichloroethane
GC/MS SIM	EPA 8260C-SIM	1,3-Butadiene
GC/MS SIM	EPA 8260C-SIM	1,4-Dichlorobenzene
GC/MS SIM	EPA 8260C-SIM	Benzene
GC/MS SIM	EPA 8260C-SIM	Bromodichloromethane
GC/MS SIM	EPA 8260C-SIM	Bromoform
GC/MS SIM	EPA 8260C-SIM	Bromomethane
GC/MS SIM	EPA 8260C-SIM	Chloroform
GC/MS SIM	EPA 8260C-SIM	Dibromochloromethane
GC/MS SIM	EPA 8260C-SIM	Hexachlorobutadiene
GC/MS SIM	EPA 8260C-SIM	Naphthalene
GC/MS SIM	EPA 8260C-SIM	Tetrachloroethene
GC/MS SIM	EPA 8260C-SIM	Trichloroethene
GC/MS SIM	EPA 8260C-SIM	Vinyl Chloride
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	1-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270D-SIM	3,3'-Dichlorobenzidine
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(b)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(g,h,i)perylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(k)fluoranthene
GC/MS SIM	EPA 8270D-SIM	Bis(2-chloroethyl) Ether
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Chrysene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluorene
GC/MS SIM	EPA 8270D-SIM	Hexachlorobenzene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Naphthalene
GC/MS SIM	EPA 8270D-SIM	n-Nitrosodimethylamine
GC/MS SIM	EPA 8270D-SIM	n-Nitrosodi-n-propylamine
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Phenanthrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Pyrene
GC/MS SIM	EPA 8270C-SIM Modified / WS-MS-0011	1,4-Dioxane
GC-IT/MS	EPA 521 Modified / WS-MS-0012	N-Nitrosodimethyl amine (NDMA)
GC-FID	EPA 8015B/8015C/8015D AK102	Diesel Range Organics (DRO)
GC-FID	AK103	Residual Range Organics
GC-FID	EPA 8015B/8015C/8015D	Motor Oil Range Organics (MRO)
GC-ECD	EPA 8081A/8081B	Aldrin
GC-ECD	EPA 8081A/8081B	a-BHC
GC-ECD	EPA 8081A/8081B	b-BHC
GC-ECD	EPA 8081A/8081B	d-BHC
GC-ECD	EPA 8081A/8081B	g-BHC (Lindane)
GC-ECD	EPA 8081A/8081B	a-Chlordane
GC-ECD	EPA 8081A/8081B	g-Chlordane
GC-ECD	EPA 8081A/8081B	4,4'-DDD
GC-ECD	EPA 8081A/8081B	4,4'-DDE
GC-ECD	EPA 8081A/8081B	4,4'-DDT
GC-ECD	EPA 8081A/8081B	Dieldrin
GC-ECD	EPA 8081A/8081B	Endosulfan I
GC-ECD	EPA 8081A/8081B	Endosulfan II
GC-ECD	EPA 8081A/8081B	Endosulfan sulfate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC-ECD	EPA 8081A/8081B	Endrin
GC-ECD	EPA 8081A/8081B	Endrin Aldehyde
GC-ECD	EPA 8081A/8081B	Endrin Ketone
GC-ECD	EPA 8081A/8081B	Heptachlor
GC-ECD	EPA 8081A/8081B	Heptachlor Epoxide
GC-ECD	EPA 8081A/8081B	Methoxychlor
GC-ECD	EPA 8081A/8081B	Toxaphene
GC-ECD	EPA 8081A/8081B	Chlordane (technical)
GC-ECD	EPA 8082/8082A	PCB-1016
GC-ECD	EPA 8082/8082A	PCB-1221
GC-ECD	EPA 8082/8082A	PCB-1232
GC-ECD	EPA 8082/8082A	PCB-1242
GC-ECD	EPA 8082/8082A	PCB-1248
GC-ECD	EPA 8082/8082A	PCB-1254
GC-ECD	EPA 8082/8082A	PCB-1260
GC-ECD	EPA 8082/8082A	PCB-1262
GC-ECD	EPA 8082/8082A	PCB-1268
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDD
GC/MS	EPA 8280A/8280B	OCDD
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDF
GC/MS	EPA 8280A/8280B	2,3,4,7,8-PeCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDF
GC/MS	EPA 8280A/8280B	2,3,4,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8,9-HpCDF
GC/MS	EPA 8280A/8280B	OCDF
GC/MS	EPA 8280A/8280B	Total TCDD
GC/MS	EPA 8280A/8280B	Total PeCDD

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8280A/8280B	Total HxCDD
GC/MS	EPA 8280A/8280B	Total HeptaCDD
GC/MS	EPA 8280A/8280B	Total TCDF
GC/MS	EPA 8280A/8280B	Total PeCDF
GC/MS	EPA 8280A/8280B	Total HxCDF
GC/MS	EPA 8280A/8280B	Total HpCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,7,8-TeCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8-PeCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,6,7,8-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8,9-HxCDD
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,6,7,8-HpCDD
GC/HRMS	EPA 8290/8290A/1613B	OCDD
GC/HRMS	EPA 8290/8290A/1613B	2,3,7,8-TeCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8-PeCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,4,7,8-PeCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,6,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,7,8,9-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	2,3,4,6,7,8-HxCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,6,7,8-HpCDF
GC/HRMS	EPA 8290/8290A/1613B	1,2,3,4,7,8,9-HpCDF
GC/HRMS	EPA 8290/8290A/1613B	OCDF
GC/HRMS	EPA 8290/8290A/1613B	Total TCDD
GC/HRMS	EPA 8290/8290A/1613B	Total PeCDD
GC/HRMS	EPA 8290/8290A/1613B	Total HxCDD
GC/HRMS	EPA 8290/8290A/1613B	Total HpCDD
GC/HRMS	EPA 8290/8290A/1613B	Total TCDF
GC/HRMS	EPA 8290/8290A/1613B	Total PeCDF
GC/HRMS	EPA 8290/8290A/1613B	Total HxCDF
GC/HRMS	EPA 8290/8290A/1613B	Total HpCDF
HPLC/UV	EPA 8330A/8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4-Dinitrotoluene

Non-Potable Water		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A/8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	Glycerol trinitrate (Nitroglycerin)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitro- 1,3,5-triazine (Hexogen)
HPLC/UV	EPA 8330A/8330B	Methyl-2,4,6- trinitrophenylnitramine
HPLC/UV	EPA 8330A/8330B	Nitrobenzene
HPLC/UV	EPA 8330A/8330B	2-Nitrotoluene (o-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	3-Nitrotoluene (m-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	4-Nitrotoluene (p-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	Octahydro-1,3,5,7- tetranitro1,3,5,7-tetracine (Octogen)
HPLC/UV	EPA 8330A/8330B	Picric acid
HPLC/UV	EPA 8330A/8330B	Pentaerythritol Tetranitrate
HPLC/UV	EPA 8330A/8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4,6-Trinitrotoluene
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3-dinitroso-5- nitro-1,3,5,triazine (DNX)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitroso- 1,3,5-triazine (TNX)
HPLC/UV	EPA 8330A/8330B	1-Nitroso-3,5-dinitro-1,3,5- triazacyclohexane (MNX)
HPLC/UV	EPA 8330A Modified /WS-LC-0010	Nitroguanidine
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	6:2 Fluorotelomer sulfonate (6:2 FTS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	8:2 Fluorotelomer sulfonate (8:2 FTS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	N-Ethyl perfluorooctanesulfon amidacetic acid (EtFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	N-Methyl perfluorooctanesulfon amidoacetic acid (MeFOSAA)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorooctanoic acid (PFOA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorooctane Sulfonic Acid (PFOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorobutyric acid (PFBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluoropentanoic acid (PFPA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorohexanoic acid (PFHxA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorononanoic acid (PFNA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorodecanoic acid (PFDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluoroundecanoic acid (PFUDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorododecanoic acid (PFDoDA)

Non-Potable Water		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorotridecanoic acid (PFTriA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorotetradecanoic acid (PDTeA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluoroheptane Sulfonic Acid (PFHpS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorodecane Sulfonic Acid (PFDS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluoroocatane Sulfonamide (FOSA)
GC/HRMS	EPA 1668A/1668C	PCB 1
GC/HRMS	EPA 1668A/1668C	PCB 2
GC/HRMS	EPA 1668A/1668C	PCB 3
GC/HRMS	EPA 1668A/1668C	PCB 4
GC/HRMS	EPA 1668A/1668C	PCB 5
GC/HRMS	EPA 1668A/1668C	PCB 6
GC/HRMS	EPA 1668A/1668C	PCB 7
GC/HRMS	EPA 1668A/1668C	PCB 8
GC/HRMS	EPA 1668A/1668C	PCB 9
GC/HRMS	EPA 1668A/1668C	PCB 10
GC/HRMS	EPA 1668A/1668C	PCB 11
GC/HRMS	EPA 1668A/1668C	PCB 12

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 13
GC/HRMS	EPA 1668A/1668C	PCB 14
GC/HRMS	EPA 1668A/1668C	PCB 15
GC/HRMS	EPA 1668A/1668C	PCB 16
GC/HRMS	EPA 1668A/1668C	PCB 17
GC/HRMS	EPA 1668A/1668C	PCB 18
GC/HRMS	EPA 1668A/1668C	PCB 19
GC/HRMS	EPA 1668A/1668C	PCB 20
GC/HRMS	EPA 1668A/1668C	PCB 21
GC/HRMS	EPA 1668A/1668C	PCB 22
GC/HRMS	EPA 1668A/1668C	PCB 23
GC/HRMS	EPA 1668A/1668C	PCB 24
GC/HRMS	EPA 1668A/1668C	PCB 25
GC/HRMS	EPA 1668A/1668C	PCB 26
GC/HRMS	EPA 1668A/1668C	PCB 27
GC/HRMS	EPA 1668A/1668C	PCB 28
GC/HRMS	EPA 1668A/1668C	PCB 29
GC/HRMS	EPA 1668A/1668C	PCB 30
GC/HRMS	EPA 1668A/1668C	PCB 32
GC/HRMS	EPA 1668A/1668C	PCB 31
GC/HRMS	EPA 1668A/1668C	PCB 33
GC/HRMS	EPA 1668A/1668C	PCB 34
GC/HRMS	EPA 1668A/1668C	PCB 35
GC/HRMS	EPA 1668A/1668C	PCB 36
GC/HRMS	EPA 1668A/1668C	PCB 37
GC/HRMS	EPA 1668A/1668C	PCB 38
GC/HRMS	EPA 1668A/1668C	PCB 39
GC/HRMS	EPA 1668A/1668C	PCB 40
GC/HRMS	EPA 1668A/1668C	PCB 41
GC/HRMS	EPA 1668A/1668C	PCB 42
GC/HRMS	EPA 1668A/1668C	PCB 43
GC/HRMS	EPA 1668A/1668C	PCB 44
GC/HRMS	EPA 1668A/1668C	PCB 45
GC/HRMS	EPA 1668A/1668C	PCB 46
GC/HRMS	EPA 1668A/1668C	PCB 47
GC/HRMS	EPA 1668A/1668C	PCB 48

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 49
GC/HRMS	EPA 1668A/1668C	PCB 50
GC/HRMS	EPA 1668A/1668C	PCB 51
GC/HRMS	EPA 1668A/1668C	PCB 52
GC/HRMS	EPA 1668A/1668C	PCB 53
GC/HRMS	EPA 1668A/1668C	PCB 54
GC/HRMS	EPA 1668A/1668C	PCB 55
GC/HRMS	EPA 1668A/1668C	PCB 56
GC/HRMS	EPA 1668A/1668C	PCB 57
GC/HRMS	EPA 1668A/1668C	PCB 58
GC/HRMS	EPA 1668A/1668C	PCB 59
GC/HRMS	EPA 1668A/1668C	PCB 60
GC/HRMS	EPA 1668A/1668C	PCB 61
GC/HRMS	EPA 1668A/1668C	PCB 62
GC/HRMS	EPA 1668A/1668C	PCB 63
GC/HRMS	EPA 1668A/1668C	PCB 64
GC/HRMS	EPA 1668A/1668C	PCB 65
GC/HRMS	EPA 1668A/1668C	PCB 66
GC/HRMS	EPA 1668A/1668C	PCB 67
GC/HRMS	EPA 1668A/1668C	PCB 68
GC/HRMS	EPA 1668A/1668C	PCB 69
GC/HRMS	EPA 1668A/1668C	PCB 70
GC/HRMS	EPA 1668A/1668C	PCB 71
GC/HRMS	EPA 1668A/1668C	PCB 72
GC/HRMS	EPA 1668A/1668C	PCB 73
GC/HRMS	EPA 1668A/1668C	PCB 74
GC/HRMS	EPA 1668A/1668C	PCB 75
GC/HRMS	EPA 1668A/1668C	PCB 76
GC/HRMS	EPA 1668A/1668C	PCB 77
GC/HRMS	EPA 1668A/1668C	PCB 78
GC/HRMS	EPA 1668A/1668C	PCB 79
GC/HRMS	EPA 1668A/1668C	PCB 80
GC/HRMS	EPA 1668A/1668C	PCB 81
GC/HRMS	EPA 1668A/1668C	PCB 82
GC/HRMS	EPA 1668A/1668C	PCB 83
GC/HRMS	EPA 1668A/1668C	PCB 84

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 85
GC/HRMS	EPA 1668A/1668C	PCB 86
GC/HRMS	EPA 1668A/1668C	PCB 87
GC/HRMS	EPA 1668A/1668C	PCB 88
GC/HRMS	EPA 1668A/1668C	PCB 89
GC/HRMS	EPA 1668A/1668C	PCB 90
GC/HRMS	EPA 1668A/1668C	PCB 91
GC/HRMS	EPA 1668A/1668C	PCB 92
GC/HRMS	EPA 1668A/1668C	PCB 93
GC/HRMS	EPA 1668A/1668C	PCB 94
GC/HRMS	EPA 1668A/1668C	PCB 95
GC/HRMS	EPA 1668A/1668C	PCB 96
GC/HRMS	EPA 1668A/1668C	PCB 97
GC/HRMS	EPA 1668A/1668C	PCB 98
GC/HRMS	EPA 1668A/1668C	PCB 99
GC/HRMS	EPA 1668A/1668C	PCB 100
GC/HRMS	EPA 1668A/1668C	PCB 101
GC/HRMS	EPA 1668A/1668C	PCB 102
GC/HRMS	EPA 1668A/1668C	PCB 103
GC/HRMS	EPA 1668A/1668C	PCB 104
GC/HRMS	EPA 1668A/1668C	PCB 105
GC/HRMS	EPA 1668A/1668C	PCB 106
GC/HRMS	EPA 1668A/1668C	PCB 107
GC/HRMS	EPA 1668A/1668C	PCB 108
GC/HRMS	EPA 1668A/1668C	PCB 109
GC/HRMS	EPA 1668A/1668C	PCB 110
GC/HRMS	EPA 1668A/1668C	PCB 111
GC/HRMS	EPA 1668A/1668C	PCB 112
GC/HRMS	EPA 1668A/1668C	PCB 113
GC/HRMS	EPA 1668A/1668C	PCB 114
GC/HRMS	EPA 1668A/1668C	PCB 115
GC/HRMS	EPA 1668A/1668C	PCB 116
GC/HRMS	EPA 1668A/1668C	PCB 117
GC/HRMS	EPA 1668A/1668C	PCB 118
GC/HRMS	EPA 1668A/1668C	PCB 119
GC/HRMS	EPA 1668A/1668C	PCB 120

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 121
GC/HRMS	EPA 1668A/1668C	PCB 122
GC/HRMS	EPA 1668A/1668C	PCB 123
GC/HRMS	EPA 1668A/1668C	PCB 124
GC/HRMS	EPA 1668A/1668C	PCB 125
GC/HRMS	EPA 1668A/1668C	PCB 126
GC/HRMS	EPA 1668A/1668C	PCB 127
GC/HRMS	EPA 1668A/1668C	PCB 128
GC/HRMS	EPA 1668A/1668C	PCB 129
GC/HRMS	EPA 1668A/1668C	PCB 130
GC/HRMS	EPA 1668A/1668C	PCB 131
GC/HRMS	EPA 1668A/1668C	PCB 132
GC/HRMS	EPA 1668A/1668C	PCB 133
GC/HRMS	EPA 1668A/1668C	PCB 134
GC/HRMS	EPA 1668A/1668C	PCB 135
GC/HRMS	EPA 1668A/1668C	PCB 136
GC/HRMS	EPA 1668A/1668C	PCB 137
GC/HRMS	EPA 1668A/1668C	PCB 138
GC/HRMS	EPA 1668A/1668C	PCB 139
GC/HRMS	EPA 1668A/1668C	PCB 140
GC/HRMS	EPA 1668A/1668C	PCB 141
GC/HRMS	EPA 1668A/1668C	PCB 142
GC/HRMS	EPA 1668A/1668C	PCB 143
GC/HRMS	EPA 1668A/1668C	PCB 144
GC/HRMS	EPA 1668A/1668C	PCB 145
GC/HRMS	EPA 1668A/1668C	PCB 146
GC/HRMS	EPA 1668A/1668C	PCB 147
GC/HRMS	EPA 1668A/1668C	PCB 148
GC/HRMS	EPA 1668A/1668C	PCB 149
GC/HRMS	EPA 1668A/1668C	PCB 150
GC/HRMS	EPA 1668A/1668C	PCB 151
GC/HRMS	EPA 1668A/1668C	PCB 152
GC/HRMS	EPA 1668A/1668C	PCB 153
GC/HRMS	EPA 1668A/1668C	PCB 154
GC/HRMS	EPA 1668A/1668C	PCB 155
GC/HRMS	EPA 1668A/1668C	PCB 156

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 157
GC/HRMS	EPA 1668A/1668C	PCB 158
GC/HRMS	EPA 1668A/1668C	PCB 159
GC/HRMS	EPA 1668A/1668C	PCB 160
GC/HRMS	EPA 1668A/1668C	PCB 161
GC/HRMS	EPA 1668A/1668C	PCB 162
GC/HRMS	EPA 1668A/1668C	PCB 163
GC/HRMS	EPA 1668A/1668C	PCB 164
GC/HRMS	EPA 1668A/1668C	PCB 165
GC/HRMS	EPA 1668A/1668C	PCB 166
GC/HRMS	EPA 1668A/1668C	PCB 167
GC/HRMS	EPA 1668A/1668C	PCB 168
GC/HRMS	EPA 1668A/1668C	PCB 169
GC/HRMS	EPA 1668A/1668C	PCB 170
GC/HRMS	EPA 1668A/1668C	PCB 171
GC/HRMS	EPA 1668A/1668C	PCB 172
GC/HRMS	EPA 1668A/1668C	PCB 173
GC/HRMS	EPA 1668A/1668C	PCB 174
GC/HRMS	EPA 1668A/1668C	PCB 175
GC/HRMS	EPA 1668A/1668C	PCB 176
GC/HRMS	EPA 1668A/1668C	PCB 177
GC/HRMS	EPA 1668A/1668C	PCB 178
GC/HRMS	EPA 1668A/1668C	PCB 179
GC/HRMS	EPA 1668A/1668C	PCB 180
GC/HRMS	EPA 1668A/1668C	PCB 181
GC/HRMS	EPA 1668A/1668C	PCB 182
GC/HRMS	EPA 1668A/1668C	PCB 183
GC/HRMS	EPA 1668A/1668C	PCB 184
GC/HRMS	EPA 1668A/1668C	PCB 185
GC/HRMS	EPA 1668A/1668C	PCB 186
GC/HRMS	EPA 1668A/1668C	PCB 187
GC/HRMS	EPA 1668A/1668C	PCB 188
GC/HRMS	EPA 1668A/1668C	PCB 189
GC/HRMS	EPA 1668A/1668C	PCB 190
GC/HRMS	EPA 1668A/1668C	PCB 191
GC/HRMS	EPA 1668A/1668C	PCB 192

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 193
GC/HRMS	EPA 1668A/1668C	PCB 194
GC/HRMS	EPA 1668A/1668C	PCB 195
GC/HRMS	EPA 1668A/1668C	PCB 196
GC/HRMS	EPA 1668A/1668C	PCB 197
GC/HRMS	EPA 1668A/1668C	PCB 198
GC/HRMS	EPA 1668A/1668C	PCB 199
GC/HRMS	EPA 1668A/1668C	PCB 200
GC/HRMS	EPA 1668A/1668C	PCB 201
GC/HRMS	EPA 1668A/1668C	PCB 202
GC/HRMS	EPA 1668A/1668C	PCB 203
GC/HRMS	EPA 1668A/1668C	PCB 204
GC/HRMS	EPA 1668A/1668C	PCB 205
GC/HRMS	EPA 1668A/1668C	PCB 206
GC/HRMS	EPA 1668A/1668C	PCB 207
GC/HRMS	EPA 1668A/1668C	PCB 208
GC/HRMS	EPA 1668A/1668C	PCB 209
Preparation	**Method**	**Type**
Acid Digestion (Aqueous)	EPA 3005A/3010A	Inorganics
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Non-Volatile Organics
Solid Phase Extraction	EPA 3535A	Semivolatile and Non-Volatile Organics
Purge and Trap	EPA 5030B/5030C	Volatile Organic Compounds
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs
Silica Gel Cleanup	EPA 3630C	Column Cleanup

Drinking Water		
Technology	**Method**	**Analyte**
LC/MS/MS	EPA 537	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	EPA 537	Perfluoroheptanoic acid (PFHpA)
LC/MS/MS	EPA 537	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	EPA 537	Perfluorononanoic acid (PFNA)

LC/MS/MS	EPA 537	Perfluorooctanoic acid (PFOA)
LC/MS/MS	EPA 537	Perfluorooctane Sulfonic Acid(PFOS)
Preparation	**Method**	**Type**
Solid Phase Extraction	EPA 537	Perfluoro compounds in Drinking Water

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C	Aluminum
ICP-AES	EPA 6010B/6010C	Antimony
ICP-AES	EPA 6010B/6010C	Arsenic
ICP-AES	EPA 6010B/6010C	Barium
ICP-AES	EPA 6010B/6010C	Beryllium
ICP-AES	EPA 6010B/6010C	Boron
ICP-AES	EPA 6010B/6010C	Cadmium
ICP-AES	EPA 6010B/6010C	Calcium
ICP-AES	EPA 6010B/6010C	Chromium (Total)
ICP-AES	EPA 6010B/6010C	Cobalt
ICP-AES	EPA 6010B/6010C	Copper
ICP-AES	EPA 6010B/6010C	Iron
ICP-AES	EPA 6010B/6010C	Lead
ICP-AES	EPA 6010B/6010C	Magnesium
ICP-AES	EPA 6010B/6010C	Manganese
ICP-AES	EPA 6010B/6010C	Molybdenum
ICP-AES	EPA 6010B/6010C	Nickel
ICP-AES	EPA 6010B/6010C	Potassium
ICP-AES	EPA 6010B/6010C	Selenium
ICP-AES	EPA 6010B/6010C	Silver
ICP-AES	EPA 6010B/6010C	Sodium
ICP-AES	EPA 6010B/6010C	Thallium
ICP-AES	EPA 6010B/6010C	Tin
ICP-AES	EPA 6010B/6010C	Titanium
ICP-AES	EPA 6010B/6010C	Vanadium
ICP-AES	EPA 6010B/6010C	Zinc
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel
ICP-MS	EPA 6020/6020A	Phosphorus
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium
ICP-MS	EPA 6020/6020A	Strontium
ICP-MS	EPA 6020/6020A	Thallium
ICP-MS	EPA 6020/6020A	Tin
ICP-MS	EPA 6020/6020A	Titanium
ICP-MS	EPA 6020/6020A	Uranium
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
CVAAS	EPA 7471A/7471B	Mercury
Colorimetric	EPA 353.2	Nitrate
Colorimetric	EPA 353.2	Nitrate-nitrite
Colorimetric	EPA 353.2	Nitrite
Colorimetric/Hydrolysis	EPA 353.2 Modified /WS-WC-0050	Nitrocellulose
LC/MS/MS	EPA 6850	Perchlorate
Probe	EPA 9045C/9045D	pH
Ion Chromatography	EPA 9056A/300.0	Bromide
Ion Chromatography	EPA 9056A/300.0	Chloride
Ion Chromatography	EPA 9056A/300.0	Fluoride
Ion Chromatography	EPA 9056A/300.0	Sulfate
Ion Chromatography	EPA 9056A/300.0	Nitrate

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Ion Chromatography	EPA 9056A/300.0	Nitrite
Gravimetric	ASTM D2216	%Moisture
GC/MS	EPA 8260B/8260C	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/8260C	1,2-Dibromoethane
GC/MS	EPA 8260B/8260C	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/8260C	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1,3-Dichloropropane
GC/MS	EPA 8260B/8260C	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C	1-Chlorohexane
GC/MS	EPA 8260B/8260C	2,2-Dichloropropane
GC/MS	EPA 8260B/8260C	2-Butanone (MEK)
GC/MS	EPA 8260B/8260C	2-Chlorotoluene
GC/MS	EPA 8260B/8260C	2-Hexanone (MBK)
GC/MS	EPA 8260B/8260C	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA 8260B/8260C	4-Chlorotoluene
GC/MS	EPA 8260B/8260C	4-Isopropyltoluene
GC/MS	EPA 8260B/8260C	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 8260B/8260C	Acetone
GC/MS	EPA 8260B/8260C	Allyl Chloride
GC/MS	EPA 8260B/8260C	Benzene
GC/MS	EPA 8260B/8260C	Bromobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	Bromochloromethane
GC/MS	EPA 8260B/8260C	Bromodichloromethane
GC/MS	EPA 8260B/8260C	Bromoform
GC/MS	EPA 8260B/8260C	Bromomethane
GC/MS	EPA 8260B/8260C	Carbon Disulfide
GC/MS	EPA 8260B/8260C	Carbon Tetrachloride
GC/MS	EPA 8260B/8260C	Chlorobenzene
GC/MS	EPA 8260B/8260C	Chloroethane
GC/MS	EPA 8260B/8260C	Chloroform
GC/MS	EPA 8260B/8260C	Chloromethane
GC/MS	EPA 8260B/8260C	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Cyclohexane
GC/MS	EPA 8260B/8260C	Dibromochloromethane
GC/MS	EPA 8260B/8260C	Dibromomethane
GC/MS	EPA 8260B/8260C	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C	Diisopropyl Ether (DIPE)
GC/MS	EPA 8260B/8260C	Ethylbenzene
GC/MS	EPA 8260B/8260C	Ethylmethacrylate
GC/MS	EPA 8260B/8260C	Ethyl tert-butyl Ether (ETBE)
GC/MS	EPA 8260B/8260C	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C	Hexane
GC/MS	EPA 8260B/8260C	Iodomethane
GC/MS	EPA 8260B/8260C	Isobutanol (2-Methyl-1-propanol)
GC/MS	EPA 8260B/8260C	Isopropylbenzene
GC/MS	EPA 8260B/8260C	m & p Xylene
GC/MS	EPA 8260B/8260C	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA 8260B/8260C	Methylene Chloride
GC/MS	EPA 8260B/8260C	Naphthalene
GC/MS	EPA 8260B/8260C	n-Butylbenzene
GC/MS	EPA 8260B/8260C	n-Propylbenzene
GC/MS	EPA 8260B/8260C	o-Xylene
GC/MS	EPA 8260B/8260C	sec-Butylbenzene
GC/MS	EPA 8260B/8260C	Styrene
GC/MS	EPA 8260B/8260C	t-Amyl methyl Ether (TAME)
GC/MS	EPA 8260B/8260C	t-1,4-Dichloro-2-Butene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C	tert-Butylbenzene
GC/MS	EPA 8260B/8260C	Tetrachloroethene
GC/MS	EPA 8260B/8260C	Toluene
GC/MS	EPA 8260B/8260C	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C	Trichloroethene
GC/MS	EPA 8260B/8260C	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C	Vinyl Acetate
GC/MS	EPA 8260B/8260C	Vinyl Chloride
GC/MS	EPA 8260B/8260C	Xylenes, Total
GC/MS	EPA 8260B/AK101MS	Gasoline Range Organics (GRO)
GC/MS	EPA 8270C/8270D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/8270D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270C/8270D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1,3-Dinitrobenzene
GC/MS	EPA 8270C/8270D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/8270D	1-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/8270D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,4-Dimethylphenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrophenol
GC/MS	EPA 8270C/8270D	2,4-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2,6-Dichlorophenol
GC/MS	EPA 8270C/8270D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/8270D	2-Chloronaphthalene
GC/MS	EPA 8270C/8270D	2-Chlorophenol
GC/MS	EPA 8270C/8270D	2-Methylnaphthalene
GC/MS	EPA 8270C/8270D	2-Methylphenol
GC/MS	EPA 8270C/8270D	2-Nitroaniline
GC/MS	EPA 8270C/8270D	2-Nitrophenol
GC/MS	EPA 8270C/8270D	3&4-Methylphenol
GC/MS	EPA 8270C/8270D	3,3'-Dichlorobenzidine

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	3-Nitroaniline
GC/MS	EPA 8270C/8270D	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/8270D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/8270D	4-Chloroaniline
GC/MS	EPA 8270C/8270D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/8270D	4-Nitroaniline
GC/MS	EPA 8270C/8270D	4-Nitrophenol
GC/MS	EPA 8270C/8270D	Acenaphthene
GC/MS	EPA 8270C/8270D	Acenaphthylene
GC/MS	EPA 8270C/8270D	Aniline
GC/MS	EPA 8270C/8270D	Anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)anthracene
GC/MS	EPA 8270C/8270D	Benzo(a)pyrene
GC/MS	EPA 8270C/8270D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/8270D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/8270D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/8270D	Benzoic Acid
GC/MS	EPA 8270C/8270D	Benzyl Alcohol
GC/MS	EPA 8270C/8270D	Benzyl butyl Phthalate
GC/MS	EPA 8270C/8270D	Biphenyl
GC/MS	EPA 8270C/8270D	Bis(2-chloroethoxy) Methane
GC/MS	EPA 8270C/8270D	Bis(2-chloroethyl) Ether
GC/MS	EPA 8270C/8270D	Bis(2-chloroisopropyl) Ether
GC/MS	EPA 8270C/8270D	Carbazole
GC/MS	EPA 8270C/8270D	Chrysene
GC/MS	EPA 8270C/8270D	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA 8270C/8270D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/8270D	Dibenzofuran
GC/MS	EPA 8270C/8270D	Diethyl Phthalate
GC/MS	EPA 8270C/8270D	Dimethyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-butyl Phthalate
GC/MS	EPA 8270C/8270D	Di-n-octyl Phthalate
GC/MS	EPA 8270C/8270D	Fluoranthene
GC/MS	EPA 8270C/8270D	Fluorene
GC/MS	EPA 8270C/8270D	Hexachlorobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/8270D	Hexachlorobutadiene
GC/MS	EPA 8270C/8270D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/8270D	Hexachloroethane
GC/MS	EPA 8270C/8270D	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA 8270C/8270D	Isophorone
GC/MS	EPA 8270C/8270D	Naphthalene
GC/MS	EPA 8270C/8270D	Nitrobenzene
GC/MS	EPA 8270C/8270D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/8270D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/8270D	Pentachlorophenol
GC/MS	EPA 8270C/8270D	Phenanthrene
GC/MS	EPA 8270C/8270D	Phenol
GC/MS	EPA 8270C/8270D	Pyrene
GC/MS	EPA 8270C/8270D	Pyridine
GC/MS SIM	EPA 8260C-SIM	1,1,2-Trichloroethane
GC/MS SIM	EPA 8260C-SIM	1,1,2,2-Tetrachloroethane
GC/MS SIM	EPA 8260C-SIM	1,2,3-Trichloropropane
GC/MS SIM	EPA 8260C-SIM	1,2-Dibromoethane
GC/MS SIM	EPA 8260C-SIM	1,2-Dichloroethane
GC/MS SIM	EPA 8260C-SIM	1,3-Butadiene
GC/MS SIM	EPA 8260C-SIM	1,4-Dichlorobenzene
GC/MS SIM	EPA 8260C-SIM	Benzene
GC/MS SIM	EPA 8260C-SIM	Bromodichloromethane
GC/MS SIM	EPA 8260C-SIM	Bromoform
GC/MS SIM	EPA 8260C-SIM	Bromomethane
GC/MS SIM	EPA 8260C-SIM	Chloroform
GC/MS SIM	EPA 8260C-SIM	Dibromochloromethane
GC/MS SIM	EPA 8260C-SIM	Dibromomethane
GC/MS SIM	EPA 8260C-SIM	Hexachlorobutadiene
GC/MS SIM	EPA 8260C-SIM	Naphthalene
GC/MS SIM	EPA 8260C-SIM	Tetrachloroethene
GC/MS SIM	EPA 8260C-SIM	Trichloroethene
GC/MS SIM	EPA 8260C-SIM	Vinyl Chloride
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	1-Methylnaphthalene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Acenaphthylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(a)pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(b)fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(g,h,i)perylene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Benzo(k)fluoranthene
GC/MS SIM	EPA 8270D-SIM	Bis(2-chloroethyl) Ether
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Chrysene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluoranthene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Fluorene
GC/MS SIM	EPA 8270D-SIM	Hexachlorobenzene
GC/MS SIM	EPA 8270D-SIM	Hexachlorocyclopentadiene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Naphthalene
GC/MS SIM	EPA 8270D-SIM	n-Nitrosodi-n-propylamine
GC/MS SIM	EPA 8270D-SIM	Pentachlorophenol
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Phenanthrene
GC/MS SIM	EPA 8270C-SIM EPA 8270D-SIM	Pyrene
GC/MS SIM	EPA 521 Modified / WS-MS-0012	N-Nitrosodimethyl amine (NDMA)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC-FID	EPA 8015B/8015C/8015D AK102	Diesel Range Organics (DRO)
GC-FID	AK103	Residual Range Organics
GC-FID	EPA 8015B/8015C/8015D	Motor Oil Range Organics (MRO)
GC-ECD	EPA 8081A/8081B	Aldrin
GC-ECD	EPA 8081A/8081B	a-BHC
GC-ECD	EPA 8081A/8081B	b-BHC
GC-ECD	EPA 8081A/8081B	d-BHC
GC-ECD	EPA 8081A/8081B	g-BHC (Lindane)
GC-ECD	EPA 8081A/8081B	a-Chlordane
GC-ECD	EPA 8081A/8081B	g-Chlordane
GC-ECD	EPA 8081A/8081B	4,4'-DDD
GC-ECD	EPA 8081A/8081B	4,4'-DDE
GC-ECD	EPA 8081A/8081B	4,4'-DDT
GC-ECD	EPA 8081A/8081B	Dieldrin
GC-ECD	EPA 8081A/8081B	Endosulfan I
GC-ECD	EPA 8081A/8081B	Endosulfan II
GC-ECD	EPA 8081A/8081B	Endosulfan sulfate
GC-ECD	EPA 8081A/8081B	Endrin
GC-ECD	EPA 8081A/8081B	Endrin Aldehyde
GC-ECD	EPA 8081A/8081B	Endrin Ketone
GC-ECD	EPA 8081A/8081B	Heptachlor
GC-ECD	EPA 8081A/8081B	Heptachlor Epoxide
GC-ECD	EPA 8081A/8081B	Methoxychlor
GC-ECD	EPA 8081A/8081B	Toxaphene
GC-ECD	EPA 8081A/8081B	Chlordane (technical)
GC-ECD	EPA 8082/8082A	PCB-1016
GC-ECD	EPA 8082/8082A	PCB-1221
GC-ECD	EPA 8082/8082A	PCB-1232
GC-ECD	EPA 8082/8082A	PCB-1242
GC-ECD	EPA 8082/8082A	PCB-1248
GC-ECD	EPA 8082/8082A	PCB-1254
GC-ECD	EPA 8082/8082A	PCB-1260
GC-ECD	EPA 8082/8082A	PCB-1262
GC-ECD	EPA 8082/8082A	PCB-1268
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDD

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDD
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDD
GC/MS	EPA 8280A/8280B	OCDD
GC/MS	EPA 8280A/8280B	2,3,7,8-TeCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8-PeCDF
GC/MS	EPA 8280A/8280B	2,3,4,7,8-PeCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,7,8,9-HxCDF
GC/MS	EPA 8280A/8280B	2,3,4,6,7,8-HxCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,6,7,8-HpCDF
GC/MS	EPA 8280A/8280B	1,2,3,4,7,8,9-HpCDF
GC/MS	EPA 8280A/8280B	OCDF
GC/MS	EPA 8280A/8280B	Total TCDD
GC/MS	EPA 8280A/8280B	Total PeCDD
GC/MS	EPA 8280A/8280B	Total HxCDD
GC/MS	EPA 8280A/8280B	Total HeptaCDD
GC/MS	EPA 8280A/8280B	Total TCDF
GC/MS	EPA 8280A/8280B	Total PeCDF
GC/MS	EPA 8280A/8280B	Total HxCDF
GC/MS	EPA 8280A/8280B	Total HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,7,8-TeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8-PeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,6,7,8-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8,9-HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,6,7,8-HpCDD
GC/HRMS	EPA 8290/ 8290A/1613B	OCDD
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,7,8-TeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8-PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,4,7,8-PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,6,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,7,8,9-HxCDF

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 8290/ 8290A/1613B	2,3,4,6,7,8-HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,6,7,8-HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	1,2,3,4,7,8,9-HpCDF
GC/HRMS	EPA 8290/ 8290A/1613B	OCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total TCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total PeCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total HxCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total HpCDD
GC/HRMS	EPA 8290/ 8290A/1613B	Total TCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total PeCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total HxCDF
GC/HRMS	EPA 8290/ 8290A/1613B	Total HpCDF
HPLC/UV	EPA 8330A/8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/8330B	Glycerol trinitrate (Nitroglycerin)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitro- 1,3,5-triazine (Hexogen)
HPLC/UV	EPA 8330A/8330B	Methyl-2,4,6- trinitrophenylnitramine
HPLC/UV	EPA 8330A/8330B	Nitrobenzene
HPLC/UV	EPA 8330A/8330B	2-Nitrotoluene (o-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	3-Nitrotoluene (m-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	4-Nitrotoluene (p-Nitrotoluene)
HPLC/UV	EPA 8330A/8330B	Octahydro-1,3,5,7- tetranitro1,3,5,7-tetracine (Octogen)
HPLC/UV	EPA 8330A/8330B	Picric acid
HPLC/UV	EPA 8330A/8330B	Pentaerythritol Tetranitrate
HPLC/UV	EPA 8330A/8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/8330B	2,4,6-Trinitrotoluene
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3-dinitroso-5- nitro-1,3,5,triazine (DNX)
HPLC/UV	EPA 8330A/8330B	Hexahydro-1,3,5-trinitroso- 1,3,5-triazine (TNX)
HPLC/UV	EPA 8330A/8330B	1-Nitroso-3,5-dinitro-1,3,5- triazacyclohexane (MNX)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A Modified / WS-LC-0010	Nitroguanidine
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	6:2 Fluorotelomer sulfonate (6:2 FTS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	8:2 Fluorotelomer sulfonate (8:2 FTS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	N-Ethyl perfluorooctanesulfon amidacetic acid (EtFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	N-Methyl perfluorooctanesulfon amidoacetic acide (MeFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorooctanoic acid (PFOA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorooctane Sulfonic Acid (PFOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorobutyric acid (PFBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluoropentanoic acid (PFPA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorohexanoic acid (PFHxA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluoroheptanoic acid (PFHpA)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorononanoic acid (PFNA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorodecanoic acid (PFDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluoroundecanoic acid (PFUDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorododecanoic acid (PFDoDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorotridecanoic acid (PFTriA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorotetradecanoic acid (PDTeA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorobutane Sulfonic Acid (PFBS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorohexane Sulfonic Acid (PFHxS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluoroheptane Sulfonic Acid (PFHpS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluorodecane Sulfonic Acid (PFDS)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 WS-LC-0025	Perfluoroocatane Sulfonamide (FOSA)
GC/HRMS	EPA 1668A/1668C	PCB 1
GC/HRMS	EPA 1668A/1668C	PCB 2
GC/HRMS	EPA 1668A/1668C	PCB 3
GC/HRMS	EPA 1668A/1668C	PCB 4
GC/HRMS	EPA 1668A/1668C	PCB 5
GC/HRMS	EPA 1668A/1668C	PCB 6
GC/HRMS	EPA 1668A/1668C	PCB 7
GC/HRMS	EPA 1668A/1668C	PCB 8
GC/HRMS	EPA 1668A/1668C	PCB 9
GC/HRMS	EPA 1668A/1668C	PCB 10
GC/HRMS	EPA 1668A/1668C	PCB 11
GC/HRMS	EPA 1668A/1668C	PCB 12
GC/HRMS	EPA 1668A/1668C	PCB 13
GC/HRMS	EPA 1668A/1668C	PCB 14
GC/HRMS	EPA 1668A/1668C	PCB 15
GC/HRMS	EPA 1668A/1668C	PCB 16
GC/HRMS	EPA 1668A/1668C	PCB 17
GC/HRMS	EPA 1668A/1668C	PCB 18
GC/HRMS	EPA 1668A/1668C	PCB 19
GC/HRMS	EPA 1668A/1668C	PCB 20
GC/HRMS	EPA 1668A/1668C	PCB 21
GC/HRMS	EPA 1668A/1668C	PCB 22
GC/HRMS	EPA 1668A/1668C	PCB 23
GC/HRMS	EPA 1668A/1668C	PCB 24
GC/HRMS	EPA 1668A/1668C	PCB 25
GC/HRMS	EPA 1668A/1668C	PCB 26
GC/HRMS	EPA 1668A/1668C	PCB 27
GC/HRMS	EPA 1668A/1668C	PCB 28
GC/HRMS	EPA 1668A/1668C	PCB 29
GC/HRMS	EPA 1668A/1668C	PCB 30
GC/HRMS	EPA 1668A/1668C	PCB 32
GC/HRMS	EPA 1668A/1668C	PCB 31
GC/HRMS	EPA 1668A/1668C	PCB 33

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 34
GC/HRMS	EPA 1668A/1668C	PCB 35
GC/HRMS	EPA 1668A/1668C	PCB 36
GC/HRMS	EPA 1668A/1668C	PCB 37
GC/HRMS	EPA 1668A/1668C	PCB 38
GC/HRMS	EPA 1668A/1668C	PCB 39
GC/HRMS	EPA 1668A/1668C	PCB 40
GC/HRMS	EPA 1668A/1668C	PCB 41
GC/HRMS	EPA 1668A/1668C	PCB 42
GC/HRMS	EPA 1668A/1668C	PCB 43
GC/HRMS	EPA 1668A/1668C	PCB 44
GC/HRMS	EPA 1668A/1668C	PCB 45
GC/HRMS	EPA 1668A/1668C	PCB 46
GC/HRMS	EPA 1668A/1668C	PCB 47
GC/HRMS	EPA 1668A/1668C	PCB 48
GC/HRMS	EPA 1668A/1668C	PCB 49
GC/HRMS	EPA 1668A/1668C	PCB 50
GC/HRMS	EPA 1668A/1668C	PCB 51
GC/HRMS	EPA 1668A/1668C	PCB 52
GC/HRMS	EPA 1668A/1668C	PCB 53
GC/HRMS	EPA 1668A/1668C	PCB 54
GC/HRMS	EPA 1668A/1668C	PCB 55
GC/HRMS	EPA 1668A/1668C	PCB 56
GC/HRMS	EPA 1668A/1668C	PCB 57
GC/HRMS	EPA 1668A/1668C	PCB 58
GC/HRMS	EPA 1668A/1668C	PCB 59
GC/HRMS	EPA 1668A/1668C	PCB 60
GC/HRMS	EPA 1668A/1668C	PCB 61
GC/HRMS	EPA 1668A/1668C	PCB 62
GC/HRMS	EPA 1668A/1668C	PCB 63
GC/HRMS	EPA 1668A/1668C	PCB 64
GC/HRMS	EPA 1668A/1668C	PCB 65
GC/HRMS	EPA 1668A/1668C	PCB 66
GC/HRMS	EPA 1668A/1668C	PCB 67
GC/HRMS	EPA 1668A/1668C	PCB 68
GC/HRMS	EPA 1668A/1668C	PCB 69

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 70
GC/HRMS	EPA 1668A/1668C	PCB 71
GC/HRMS	EPA 1668A/1668C	PCB 72
GC/HRMS	EPA 1668A/1668C	PCB 73
GC/HRMS	EPA 1668A/1668C	PCB 74
GC/HRMS	EPA 1668A/1668C	PCB 75
GC/HRMS	EPA 1668A/1668C	PCB 76
GC/HRMS	EPA 1668A/1668C	PCB 77
GC/HRMS	EPA 1668A/1668C	PCB 78
GC/HRMS	EPA 1668A/1668C	PCB 79
GC/HRMS	EPA 1668A/1668C	PCB 80
GC/HRMS	EPA 1668A/1668C	PCB 81
GC/HRMS	EPA 1668A/1668C	PCB 82
GC/HRMS	EPA 1668A/1668C	PCB 83
GC/HRMS	EPA 1668A/1668C	PCB 84
GC/HRMS	EPA 1668A/1668C	PCB 85
GC/HRMS	EPA 1668A/1668C	PCB 86
GC/HRMS	EPA 1668A/1668C	PCB 87
GC/HRMS	EPA 1668A/1668C	PCB 88
GC/HRMS	EPA 1668A/1668C	PCB 89
GC/HRMS	EPA 1668A/1668C	PCB 90
GC/HRMS	EPA 1668A/1668C	PCB 91
GC/HRMS	EPA 1668A/1668C	PCB 92
GC/HRMS	EPA 1668A/1668C	PCB 93
GC/HRMS	EPA 1668A/1668C	PCB 94
GC/HRMS	EPA 1668A/1668C	PCB 95
GC/HRMS	EPA 1668A/1668C	PCB 96
GC/HRMS	EPA 1668A/1668C	PCB 97
GC/HRMS	EPA 1668A/1668C	PCB 98
GC/HRMS	EPA 1668A/1668C	PCB 99
GC/HRMS	EPA 1668A/1668C	PCB 100
GC/HRMS	EPA 1668A/1668C	PCB 101
GC/HRMS	EPA 1668A/1668C	PCB 102
GC/HRMS	EPA 1668A/1668C	PCB 103
GC/HRMS	EPA 1668A/1668C	PCB 104
GC/HRMS	EPA 1668A/1668C	PCB 105

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 106
GC/HRMS	EPA 1668A/1668C	PCB 107
GC/HRMS	EPA 1668A/1668C	PCB 108
GC/HRMS	EPA 1668A/1668C	PCB 109
GC/HRMS	EPA 1668A/1668C	PCB 110
GC/HRMS	EPA 1668A/1668C	PCB 111
GC/HRMS	EPA 1668A/1668C	PCB 112
GC/HRMS	EPA 1668A/1668C	PCB 113
GC/HRMS	EPA 1668A/1668C	PCB 114
GC/HRMS	EPA 1668A/1668C	PCB 115
GC/HRMS	EPA 1668A/1668C	PCB 116
GC/HRMS	EPA 1668A/1668C	PCB 117
GC/HRMS	EPA 1668A/1668C	PCB 118
GC/HRMS	EPA 1668A/1668C	PCB 119
GC/HRMS	EPA 1668A/1668C	PCB 120
GC/HRMS	EPA 1668A/1668C	PCB 121
GC/HRMS	EPA 1668A/1668C	PCB 122
GC/HRMS	EPA 1668A/1668C	PCB 123
GC/HRMS	EPA 1668A/1668C	PCB 124
GC/HRMS	EPA 1668A/1668C	PCB 125
GC/HRMS	EPA 1668A/1668C	PCB 126
GC/HRMS	EPA 1668A/1668C	PCB 127
GC/HRMS	EPA 1668A/1668C	PCB 128
GC/HRMS	EPA 1668A/1668C	PCB 129
GC/HRMS	EPA 1668A/1668C	PCB 130
GC/HRMS	EPA 1668A/1668C	PCB 131
GC/HRMS	EPA 1668A/1668C	PCB 132
GC/HRMS	EPA 1668A/1668C	PCB 133
GC/HRMS	EPA 1668A/1668C	PCB 134
GC/HRMS	EPA 1668A/1668C	PCB 135
GC/HRMS	EPA 1668A/1668C	PCB 136
GC/HRMS	EPA 1668A/1668C	PCB 137
GC/HRMS	EPA 1668A/1668C	PCB 138
GC/HRMS	EPA 1668A/1668C	PCB 139
GC/HRMS	EPA 1668A/1668C	PCB 140
GC/HRMS	EPA 1668A/1668C	PCB 141

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 142
GC/HRMS	EPA 1668A/1668C	PCB 143
GC/HRMS	EPA 1668A/1668C	PCB 144
GC/HRMS	EPA 1668A/1668C	PCB 145
GC/HRMS	EPA 1668A/1668C	PCB 146
GC/HRMS	EPA 1668A/1668C	PCB 147
GC/HRMS	EPA 1668A/1668C	PCB 148
GC/HRMS	EPA 1668A/1668C	PCB 149
GC/HRMS	EPA 1668A/1668C	PCB 150
GC/HRMS	EPA 1668A/1668C	PCB 151
GC/HRMS	EPA 1668A/1668C	PCB 152
GC/HRMS	EPA 1668A/1668C	PCB 153
GC/HRMS	EPA 1668A/1668C	PCB 154
GC/HRMS	EPA 1668A/1668C	PCB 155
GC/HRMS	EPA 1668A/1668C	PCB 156
GC/HRMS	EPA 1668A/1668C	PCB 157
GC/HRMS	EPA 1668A/1668C	PCB 158
GC/HRMS	EPA 1668A/1668C	PCB 159
GC/HRMS	EPA 1668A/1668C	PCB 160
GC/HRMS	EPA 1668A/1668C	PCB 161
GC/HRMS	EPA 1668A/1668C	PCB 162
GC/HRMS	EPA 1668A/1668C	PCB 163
GC/HRMS	EPA 1668A/1668C	PCB 164
GC/HRMS	EPA 1668A/1668C	PCB 165
GC/HRMS	EPA 1668A/1668C	PCB 166
GC/HRMS	EPA 1668A/1668C	PCB 167
GC/HRMS	EPA 1668A/1668C	PCB 168
GC/HRMS	EPA 1668A/1668C	PCB 169
GC/HRMS	EPA 1668A/1668C	PCB 170
GC/HRMS	EPA 1668A/1668C	PCB 171
GC/HRMS	EPA 1668A/1668C	PCB 172
GC/HRMS	EPA 1668A/1668C	PCB 173
GC/HRMS	EPA 1668A/1668C	PCB 174
GC/HRMS	EPA 1668A/1668C	PCB 175
GC/HRMS	EPA 1668A/1668C	PCB 176
GC/HRMS	EPA 1668A/1668C	PCB 177

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/HRMS	EPA 1668A/1668C	PCB 178
GC/HRMS	EPA 1668A/1668C	PCB 179
GC/HRMS	EPA 1668A/1668C	PCB 180
GC/HRMS	EPA 1668A/1668C	PCB 181
GC/HRMS	EPA 1668A/1668C	PCB 182
GC/HRMS	EPA 1668A/1668C	PCB 183
GC/HRMS	EPA 1668A/1668C	PCB 184
GC/HRMS	EPA 1668A/1668C	PCB 185
GC/HRMS	EPA 1668A/1668C	PCB 186
GC/HRMS	EPA 1668A/1668C	PCB 187
GC/HRMS	EPA 1668A/1668C	PCB 188
GC/HRMS	EPA 1668A/1668C	PCB 189
GC/HRMS	EPA 1668A/1668C	PCB 190
GC/HRMS	EPA 1668A/1668C	PCB 191
GC/HRMS	EPA 1668A/1668C	PCB 192
GC/HRMS	EPA 1668A/1668C	PCB 193
GC/HRMS	EPA 1668A/1668C	PCB 194
GC/HRMS	EPA 1668A/1668C	PCB 195
GC/HRMS	EPA 1668A/1668C	PCB 196
GC/HRMS	EPA 1668A/1668C	PCB 197
GC/HRMS	EPA 1668A/1668C	PCB 198
GC/HRMS	EPA 1668A/1668C	PCB 199
GC/HRMS	EPA 1668A/1668C	PCB 200
GC/HRMS	EPA 1668A/1668C	PCB 201
GC/HRMS	EPA 1668A/1668C	PCB 202
GC/HRMS	EPA 1668A/1668C	PCB 203
GC/HRMS	EPA 1668A/1668C	PCB 204
GC/HRMS	EPA 1668A/1668C	PCB 205
GC/HRMS	EPA 1668A/1668C	PCB 206
GC/HRMS	EPA 1668A/1668C	PCB 207
GC/HRMS	EPA 1668A/1668C	PCB 208
GC/HRMS	EPA 1668A/1668C	PCB 209
Preparation	**Method**	**Type**
Acid Digestion (Aqueous)	EPA 3005A/3010A	Inorganics
Acid Digestion (Solid)	EPA 3050B	Inorganics
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Non-Volatile Organics

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Ultrasonic Extraction	EPA 3550B/3550C	Semivolatile and Non-Volatile Organics
Solvent Dilution	EPA 3580A	Semivolatile and Non-Volatile Organics
Purge and Trap	EPA 5030B	Volatile Organic Compounds
Purge and Trap	EPA 5035/5035A	Volatile Organic Compounds
Microwave Extraction	EPA 3546	Semivolatile and Non-Volatile Organics
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs
Silica Gel Cleanup	EPA 3630C	Column Cleanup
TCLP Extraction	EPA 1311	Toxicity Characteristic Leaching Procedure

Air and Emissions		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A	Aluminum
ICP-MS	EPA 6020/6020A	Antimony
ICP-MS	EPA 6020/6020A	Arsenic
ICP-MS	EPA 6020/6020A	Barium
ICP-MS	EPA 6020/6020A	Beryllium
ICP-MS	EPA 6020/6020A	Cadmium
ICP-MS	EPA 6020/6020A	Calcium
ICP-MS	EPA 6020/6020A	Chromium (Total)
ICP-MS	EPA 6020/6020A	Cobalt
ICP-MS	EPA 6020/6020A	Copper
ICP-MS	EPA 6020/6020A	Iron
ICP-MS	EPA 6020/6020A	Lead
ICP-MS	EPA 6020/6020A	Magnesium
ICP-MS	EPA 6020/6020A	Manganese
ICP-MS	EPA 6020/6020A	Molybdenum
ICP-MS	EPA 6020/6020A	Nickel
ICP-MS	EPA 6020/6020A	Potassium
ICP-MS	EPA 6020/6020A	Selenium
ICP-MS	EPA 6020/6020A	Silver
ICP-MS	EPA 6020/6020A	Sodium
ICP-MS	EPA 6020/6020A	Thallium

Air and Emissions		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A	Vanadium
ICP-MS	EPA 6020/6020A	Zinc
Gravimetric	40CFR Part 50 App B	TSP (Total Suspended Particulate)
Gravimetric	40CFR Part 50 App J	PM10
GC/MS	EPA TO-14A/TO-15	1,1,1-Trichloroethane
GC/MS	EPA TO-14A/TO-15	1,1,2,2-Tetrachloroethane
GC/MS	EPA TO-14A/TO-15	1,1,2-Trichloroethane
GC/MS	EPA TO-14A/TO-15	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA TO-14A/TO-15	1,1-Dichloroethane
GC/MS	EPA TO-14A/TO-15	1,1-Dichloroethene
GC/MS	EPA TO-14A/TO-15	1,2,3-Trichlorobenzene
GC/MS	EPA TO-14A/TO-15	1,2,3-Trichloropropane
GC/MS	EPA TO-14A/TO-15	1,2,4-Trichlorobenzene
GC/MS	EPA TO-14A/TO-15	1,2,4-Trimethylbenzene
GC/MS	EPA TO-14A/TO-15	1,2-Dibromoethane
GC/MS	EPA TO-14A/TO-15	1,2-Dichlorobenzene
GC/MS	EPA TO-14A/TO-15	1,2-Dichloroethane
GC/MS	EPA TO-14A/TO-15	1,2-Dichloropropane
GC/MS	EPA TO-14A/TO-15	1,3,5-Trimethylbenzene
GC/MS	EPA TO-14A/TO-15	1,3-Dichlorobenzene
GC/MS	EPA TO-14A/TO-15	1,4-Dichlorobenzene
GC/MS	EPA TO-14A/TO-15	1,4-Dioxane
GC/MS	EPA TO-14A/TO-15	2-Butanone (MEK)
GC/MS	EPA TO-14A/TO-15	2-Chlorotoluene
GC/MS	EPA TO-14A/TO-15	2-Hexanone (MBK)
GC/MS	EPA TO-14A/TO-15	2-Methyl-2-propanol (tert- Butyl Alcohol, TBA)
GC/MS	EPA TO-14A/TO-15	4-Ethyltoluene
GC/MS	EPA TO-14A/TO-15	4-Isopropyltoluene
GC/MS	EPA TO-14A/TO-15	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA TO-14A/TO-15	Acetone
GC/MS	EPA TO-14A/TO-15	Acrolein
GC/MS	EPA TO-14A/TO-15	Allyl Chloride
GC/MS	EPA TO-14A/TO-15	Alpha Methyl Styrene
GC/MS	EPA TO-14A/TO-15	Benzene
GC/MS	EPA TO-14A/TO-15	Benzyl Chloride
GC/MS	EPA TO-14A/TO-15	Bromodichloromethane

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-14A/TO-15	Bromoform
GC/MS	EPA TO-14A/TO-15	Bromomethane
GC/MS	EPA TO-14A/TO-15	Butadiene (1,3-Butadiene)
GC/MS	EPA TO-14A/TO-15	Butane
GC/MS	EPA TO-14A/TO-15	Carbon Disulfide
GC/MS	EPA TO-14A/TO-15	Carbon Tetrachloride
GC/MS	EPA TO-14A/TO-15	Chlorobenzene
GC/MS	EPA TO-14A/TO-15	Chlorodifluoromethane
GC/MS	EPA TO-14A/TO-15	Chloroethane
GC/MS	EPA TO-14A/TO-15	Chloroform
GC/MS	EPA TO-14A/TO-15	Chloromethane
GC/MS	EPA TO-14A/TO-15	cis-1,2-Dichloroethene
GC/MS	EPA TO-14A/TO-15	cis-1,3-Dichloropropene
GC/MS	EPA TO-14A/TO-15	Cyclohexane
GC/MS	EPA TO-14A/TO-15	Dibromochloromethane
GC/MS	EPA TO-14A/TO-15	Dibromomethane
GC/MS	EPA TO-14A/TO-15	Dichlorodifluoromethane
GC/MS	EPA TO-14A/TO-15	Ethyl Acetate
GC/MS	EPA TO-14A/TO-15	Ethylbenzene
GC/MS	EPA TO-14A/TO-15	Hexachlorobutadiene
GC/MS	EPA TO-14A/TO-15	Hexane
GC/MS	EPA TO-14A/TO-15	Isooctane (2,2,4- Trimethylpentane)
GC/MS	EPA TO-14A/TO-15	Isopropyl Alcohol
GC/MS	EPA TO-14A/TO-15	Isopropylbenzene
GC/MS	EPA TO-14A/TO-15	m & p Xylene
GC/MS	EPA TO-14A/TO-15	Methyl tert-butyl Ether (MTBE)
GC/MS	EPA TO-14A/TO-15	Methylene Chloride
GC/MS	EPA TO-14A/TO-15	Naphthalene
GC/MS	EPA TO-14A/TO-15	n-Butanol
GC/MS	EPA TO-14A/TO-15	n-Butylbenzene
GC/MS	EPA TO-14A/TO-15	n-Heptane
GC/MS	EPA TO-14A/TO-15	n-Nonane
GC/MS	EPA TO-14A/TO-15	n-Octane
GC/MS	EPA TO-14A/TO-15	n-Propylbenzene
GC/MS	EPA TO-14A/TO-15	o-Xylene
GC/MS	EPA TO-14A/TO-15	Pentane

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-14A/TO-15	Propene
GC/MS	EPA TO-14A/TO-15	sec-Butylbenzene
GC/MS	EPA TO-14A/TO-15	Styrene
GC/MS	EPA TO-14A/TO-15	tert-Butylbenzene
GC/MS	EPA TO-14A/TO-15	Tetrachloroethene
GC/MS	EPA TO-14A/TO-15	Tetrahydrofuran
GC/MS	EPA TO-14A/TO-15	Toluene
GC/MS	EPA TO-14A/TO-15	trans-1,2-Dichloroethene
GC/MS	EPA TO-14A/TO-15	trans-1,3-Dichloropropene
GC/MS	EPA TO-14A/TO-15	Trichloroethene
GC/MS	EPA TO-14A/TO-15	Trichlorofluoromethane
GC/MS	EPA TO-14A/TO-15	Vinyl Acetate
GC/MS	EPA TO-14A/TO-15	Vinyl Bromide
GC/MS	EPA TO-14A/TO-15	Vinyl Chloride
GC/MS	EPA TO-14A/TO-15	Xylenes, Total
GC-FID/TCD	ASTM1946D / EPA 3C	Carbon Dioxide
GC-FID/TCD	ASTM1946D / EPA 3C	Nitrogen
GC-FID/TCD	ASTM1946D / EPA 3C	Oxygen
GC-FID/TCD	ASTM1946D / EPA 3C	Helium
GC-FID/TCD	ASTM1946D / EPA 3C	Hydrogen
GC-FID/TCD	ASTM1946D / EPA 3C	Methane
GC-FID/TCD	ASTM1946D / EPA 3C	Carbon Monoxide
GC/MS	EPA TO-14A/TO-15	Gasoline Range Organics (GRO)
GC/MS	EPA TO-14A/TO-15	TPH as Gasoline
GC/MS SIM	EPA TO-15 SIM	1,1,1-Trichloroethane
GC/MS SIM	EPA TO-15 SIM	1,1,2,2-Tetrachloroethane
GC/MS SIM	EPA TO-15 SIM	1,1,2-Trichloroethane
GC/MS SIM	EPA TO-15 SIM	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS SIM	EPA TO-15 SIM	1,1-Dichloroethane
GC/MS SIM	EPA TO-15 SIM	1,1-Dichloroethene
GC/MS SIM	EPA TO-15 SIM	1,2,3-Trichloropropane
GC/MS SIM	EPA TO-15 SIM	1,2,4-Trichlorobenzene
GC/MS SIM	EPA TO-15 SIM	1,2-Dibromoethane
GC/MS SIM	EPA TO-15 SIM	1,2-Dichlorobenzene
GC/MS SIM	EPA TO-15 SIM	1,2-Dichloroethane
GC/MS SIM	EPA TO-15 SIM	1,2-Dichloropropane

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA TO-15 SIM	1,3-Dichlorobenzene
GC/MS SIM	EPA TO-15 SIM	1,4-Dichlorobenzene
GC/MS SIM	EPA TO-15 SIM	1,4-Dioxane
GC/MS SIM	EPA TO-15 SIM	Acrolein
GC/MS SIM	EPA TO-15 SIM	Benzene
GC/MS SIM	EPA TO-15 SIM	Benzyl Chloride
GC/MS SIM	EPA TO-15 SIM	Bromodichloromethane
GC/MS SIM	EPA TO-15 SIM	Butadiene (1,3-Butadiene)
GC/MS SIM	EPA TO-15 SIM	Carbon Tetrachloride
GC/MS SIM	EPA TO-15 SIM	Chlorobenzene
GC/MS SIM	EPA TO-15 SIM	Chloroethane
GC/MS SIM	EPA TO-15 SIM	Chloroform
GC/MS SIM	EPA TO-15 SIM	Chloromethane
GC/MS SIM	EPA TO-15 SIM	cis-1,2-Dichloroethene
GC/MS SIM	EPA TO-15 SIM	cis-1,3-Dichloropropene
GC/MS SIM	EPA TO-15 SIM	Dibromochloromethane
GC/MS SIM	EPA TO-15 SIM	Dichlorodifluoromethane
GC/MS SIM	EPA TO-15 SIM	Ethylbenzene
GC/MS SIM	EPA TO-15 SIM	Hexachlorobutadiene
GC/MS SIM	EPA TO-15 SIM	m & p Xylene
GC/MS SIM	EPA TO-15 SIM	Methyl tert-butyl Ether (MTBE)
GC/MS SIM	EPA TO-15 SIM	Methylene Chloride
GC/MS SIM	EPA TO-15 SIM	Naphthalene
GC/MS SIM	EPA TO-15 SIM	o-Xylene
GC/MS SIM	EPA TO-15 SIM	Styrene
GC/MS SIM	EPA TO-15 SIM	Tetrachloroethene
GC/MS SIM	EPA TO-15 SIM	Toluene
GC/MS SIM	EPA TO-15 SIM	trans-1,2-Dichloroethene
GC/MS SIM	EPA TO-15 SIM	trans-1,3-Dichloropropene
GC/MS SIM	EPA TO-15 SIM	Trichloroethene
GC/MS SIM	EPA TO-15 SIM	Trichlorofluoromethane
GC/MS SIM	EPA TO-15 SIM	Vinyl Chloride
GC/MS SIM	EPA TO-15 SIM	Xylenes, Total
GC/MS	EPA TO-13A	1,2,4-Trichlorobenzene
GC/MS	EPA TO-13A	1,2-Dichlorobenzene
GC/MS	EPA TO-13A	1,3-Dichlorobenzene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-13A	1,3-Dinitrobenzene
GC/MS	EPA TO-13A	1,4-Dichlorobenzene
GC/MS	EPA TO-13A	1-Methylnaphthalene
GC/MS	EPA TO-13A	2,3,4,6-Tetrachlorophenol
GC/MS	EPA TO-13A	2,4,5-Trichlorophenol
GC/MS	EPA TO-13A	2,4,6-Trichlorophenol
GC/MS	EPA TO-13A	2,4-Dichlorophenol
GC/MS	EPA TO-13A	2,4-Dimethylphenol
GC/MS	EPA TO-13A	2,4-Dinitrophenol
GC/MS	EPA TO-13A	2,4-Dinitrotoluene
GC/MS	EPA TO-13A	2,6-Dichlorophenol
GC/MS	EPA TO-13A	2,6-Dinitrotoluene
GC/MS	EPA TO-13A	2-Chloronaphthalene
GC/MS	EPA TO-13A	2-Chlorophenol
GC/MS	EPA TO-13A	2-Methylnaphthalene
GC/MS	EPA TO-13A	2-Methylphenol
GC/MS	EPA TO-13A	2-Nitroaniline
GC/MS	EPA TO-13A	2-Nitrophenol
GC/MS	EPA TO-13A	3&4-Methylphenol
GC/MS	EPA TO-13A	3,3'-Dichlorobenzidine
GC/MS	EPA TO-13A	3-Nitroaniline
GC/MS	EPA TO-13A	4,6-Dinitro-2-methylphenol
GC/MS	EPA TO-13A	4-Bromophenyl phenyl ether
GC/MS	EPA TO-13A	4-Chloro-3-methylphenol
GC/MS	EPA TO-13A	4-Chloroaniline
GC/MS	EPA TO-13A	4-Chlorophenyl phenyl ether
GC/MS	EPA TO-13A	4-Nitroaniline
GC/MS	EPA TO-13A	4-Nitrophenol
GC/MS	EPA TO-13A	Acenaphthene
GC/MS	EPA TO-13A	Acenaphthylene
GC/MS	EPA TO-13A	Aniline
GC/MS	EPA TO-13A	Anthracene
GC/MS	EPA TO-13A	Benzo(a)anthracene
GC/MS	EPA TO-13A	Benzo(a)pyrene
GC/MS	EPA TO-13A	Benzo(b)fluoranthene
GC/MS	EPA TO-13A	Benzo(g,h,i)perylene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-13A	Benzo(k)fluoranthene
GC/MS	EPA TO-13A	Benzoic Acid
GC/MS	EPA TO-13A	Benzyl Alcohol
GC/MS	EPA TO-13A	Benzyl butyl Phthalate
GC/MS	EPA TO-13A	Biphenyl
GC/MS	EPA TO-13A	Bis(2-chloroethoxy) Methane
GC/MS	EPA TO-13A	Bis(2-chloroethyl) Ether
GC/MS	EPA TO-13A	Bis(2-chloroisopropyl) Ether
GC/MS	EPA TO-13A	Carbazole
GC/MS	EPA TO-13A	Chrysene
GC/MS	EPA TO-13A	Bis (2-ethylhexyl) Phthalate
GC/MS	EPA TO-13A	Dibenz(a,h)anthracene
GC/MS	EPA TO-13A	Dibenzofuran
GC/MS	EPA TO-13A	Diethyl Phthalate
GC/MS	EPA TO-13A	Dimethyl Phthalate
GC/MS	EPA TO-13A	Di-n-butyl Phthalate
GC/MS	EPA TO-13A	Di-n-octyl Phthalate
GC/MS	EPA TO-13A	Fluoranthene
GC/MS	EPA TO-13A	Fluorene
GC/MS	EPA TO-13A	Hexachlorobenzene
GC/MS	EPA TO-13A	Hexachlorobutadiene
GC/MS	EPA TO-13A	Hexachlorocyclopentadiene
GC/MS	EPA TO-13A	Hexachloroethane
GC/MS	EPA TO-13A	Indeno(1,2,3-c,d) Pyrene
GC/MS	EPA TO-13A	Isophorone
GC/MS	EPA TO-13A	Naphthalene
GC/MS	EPA TO-13A	Nitrobenzene
GC/MS	EPA TO-13A	n-Nitrosodimethylamine
GC/MS	EPA TO-13A	n-Nitrosodi-n-propylamine
GC/MS	EPA TO-13A	n-Nitrosodiphenylamine
GC/MS	EPA TO-13A	Pentachlorophenol
GC/MS	EPA TO-13A	Phenanthrene
GC/MS	EPA TO-13A	Phenol
GC/MS	EPA TO-13A	Pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	1-Methylnaphthalene

Air and Emissions		
Technology	**Method**	**Analyte**
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	2-Methylnaphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Acenaphthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Acenaphthylene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Anthracene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(a)anthracene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(a)pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(b)fluoranthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(g,h,i)perylene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Benzo(k)fluoranthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Chrysene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Fluoranthene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Fluorene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Indeno(1,2,3-c,d) Pyrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Naphthalene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Phenanthrene
GC/MS SIM	EPA TO-13A SIM / WS-MS-0006	Pyrene
GC-ECD	EPA TO-4A/TO-10A	PCB-1016
GC-ECD	EPA TO-4A/TO-10A	PCB-1221
GC-ECD	EPA TO-4A/TO-10A	PCB-1232
GC-ECD	EPA TO-4A/TO-10A	PCB-1242
GC-ECD	EPA TO-4A/TO-10A	PCB-1248
GC-ECD	EPA TO-4A/TO-10A	PCB-1254
GC-ECD	EPA TO-4A/TO-10A	PCB-1260

Air and Emissions		
Technology	**Method**	**Analyte**
GC-ECD	EPA TO-4A/TO-10A	PCB-1262
GC-ECD	EPA TO-4A/TO-10A	PCB-1268
Preparation	**Method**	**Type**
Acid Digestion (Filters, Solid)	EPA 3050B	Inorganics
Soxhlet extraction of PUF	TO-4A/TO-10A	PCBs in Air
Soxhlet extraction of PUF/XAD	TO-13	Semivolatiles in Air
Florisil Cleanup	EPA 3620B/3620C	Cleanup of pesticide residues and other chlorinated hydrocarbons
Sulfur Cleanup	EPA 3660A	Sulfur Cleanup
Sulfuric Acid Cleanup	EPA 3665A	Sulfuric Acid Cleanup for PCBs

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2468

Vice President

Title: Per- and Polyfluorinated Substances (PFAS) in Water, Soils, Sediments and Tissue

[Method 537 (Modified), Method PFAS by LCMSMS Compliant with QSM 5.1 Table B-15]

Approvals (Signature/Date):

Robert Hrabak Technical Manager	Date 8/17/18
Joe Schairer Health & Safety Manager / Coordinator	Date 8/17/18
Lisa Stafford Quality Assurance Manager	Date 8/17/18
Chris Williams Laboratory Manager	Date 8/10/18

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water, soil, sediment, and tissue samples for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS).

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
Perfluoro-n-hexanoic acid	PFHxA	307-24-4
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluoro-n-decanoic acid	PFDA	335-76-2
Perfluoro-n-undecanoic acid	PFUdA (PFUnA)	2058-94-8
Perfluoro-n-dodecanoic acid	PFDoA	307-55-1
Perfluoro-n-tridecanoic acid	PFTrDA	72629-94-8
Perfluoro-n-tetradecanoic acid	PFTeDA (PFTA)	376-06-7
Perfluoro-n-hexadecanoic acid (non-routine analyte)	PFHxDA	67905-19-5
Perfluoro-n-octadecanoic acid (non-routine analyte)	PFODA	16517-11-6
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
Perfluoro-1-pentanesulfonic acid	PFPeS	2706-91-1
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
Perfluoro-1-heptanesulfonic acid	PFHpS	375-92-8
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1
Perfluoro-nonanesulfonic acid	PFNS	8789-57-2
Perfluoro-1-decanesulfonic acid	PFDS	335-77-3
Perfluoro-1-dodecansulfonic acid	PFDoS	79780-39-5
Perfluorinated sulfonamides (FOSA)		
Perfluoro-1-octanesulfonamide	FOSA	754-91-6
Perfluorinated sulfonamidoacetic acids (FOSAA)		
N-ethylperfluoro-1-octanesulfonamidoacetic acid	EtFOSAA	2991-50-6
N-methylperfluoro-1-octanesulfonamidoacetic acid	MeFOSAA	2355-31-9
Fluorotelomer sulfonates (FTS)		
1H,1H,2H,2H-perfluorohexane sulfonate *(4:2)*	4:2 FTS	757124-72-4
1H,1H,2H,2H-perfluorooctane sulfonate *(6:2)*	6:2 FTS	27619-97-2
1H,1H,2H,2H-perfluorodecane sulfonate *(8:2)*	8:2 FTS	39108-34-4
1H,1H,2H,2H-perfluorododecane sulfonate *(10:2)*	10:2 FTS	120226-60-0

Abbreviations in parenthesis are the abbreviations listed in Method 537, where they differ from the abbreviation used by the laboratory's LIMS.

1.2. Additional analytes supported by this method: The following analytes can be supported by this method under special request.

Compound Name	Abbreviation	CAS #
Fluorinated Replacement Chemicals		
Dona (Donic acid)	Dona	919005-14-4
Perfluoro(2-propoxypropanoic) acid	HFPO-DA or GenX	13252-13-6
F53B (reported as the summation of the following)	F53B	NA
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonate	F53B major	73606-19-6
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonate	F5B minor	83329-89-9

1.3. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	250 mL	2.0 ng/L – 20 ng/L	2.0 ng/L - 400 ng/L
Soil/Sediment	5 g	0.2 ug/kg – 2.0 ug/kg	0.2 ug/kg - 40 ug/kg
Tissue	1 g	1.0 ug/kg – 10 ug/kg	1.0 ug/kg – 200 ug/kg

1.4. The procedure for the analysis of water samples via in line solid phase extraction (SPE) for a subset of the list in Section 1.1 using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a SCIEX 5500 is described in Attachment 1 of this SOP.

1.5. This procedure also includes direction for preparing and analyzing samples to determine "Total Oxidizable Precursors", which may assist in improving understanding of potential PFAS environmental risk.

1.6. When undertaking projects for the Department of Defense (DoD) and/or the Department of Energy (DOE) the relevant criteria in QA Policy WS-PQA-021, "Federal Program Requirements" must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. Water samples are extracted using a solid phase extraction (SPE) cartridge. PFAS are eluted from the cartridge with an ammonium hydroxide/methanol solution.

2.2. Soil/sediment/tissue samples are extracted with a KOH/methanol solution using an orbital shaker for 3 hours followed by sonication for 12 hours. The mixture is centrifuged and the solvent filtered.

2.3. The final 80:20 methanol:water extracts are analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using 20 mM ammonium acetate/water and methanol. The mass spectrometer detector is operated in the electrospray (ESI) negative ion mode for the analysis of PFAS.

2.4. An isotope dilution technique is employed with this method for the compounds of interest. The isotope dilution analytes (IDA) consist of carbon-13 labeled analogs, oxygen-18 labeled analogs, or deuterated analogs of the compounds of interest, and they are spiked into the samples at the time of extraction. This technique allows for the correction for analytical bias encountered when analyzing more chemically complex environmental samples. The isotopically labeled compounds are chemically similar to the compounds of concern and are therefore affected by sample-related interferences to the same extent as the compounds of concern. Compounds that do not have an identically labeled analog are quantitated by the IDA method using a closely related labeled analog.

2.5. Quantitation by the internal standard method is employed for the IDA analytes/recoveries. Peak response is measured as the area of the peak.

2.6. Samples for the "Total Oxidizable Precursor" assay (TOP) are analyzed in two phases – an aliquot is prepared and analyzed as a normal sample, and a second aliquot is subjected to oxidation with potassium persulfate and sodium hydroxide prior to solid phase extraction and analysis. The total perfluorocarboxylic acid value is determined for each aliquot, and the difference calculated.

3. DEFINITIONS

3.1. PFCAs: Perfluorocarboxylic acids

3.2. PFSAs: Perfluorinated sulfonic acids

3.3. FOSA: Perfluorinated sulfonamide

3.4. PFOA: Perfluorooctanoic acid

3.5. PFOS: Perfluorooctane sulfonic acid

3.6. MPFOA: Perfluoro-n-[1,2,3,4-13C4]octanoic acid. Carbon-13 labeled PFOA

3.7. MPFOS: Perfluoro-1-[1,2,3,4-13C4]octanesulfonic acid. Carbon-13 labeled PFOS

3.8. PTFE: Polytetrafluoroethylene (e.g., Teflon®)

3.9. SPE: Solid phase extraction

3.10. PP: Polypropylene

3.11. PE: Polyethylene

3.12. HDPE: High density polyethylene

3.13. AFFF: Aqueous Film Forming Foam

3.14. IDA: Isotope dilution analyte

3.15. Further definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Manual (QAM).

4. INTERFERENCES

4.1. PFAS have been used in a wide variety of manufacturing processes, and laboratory supplies should be considered potentially contaminated until they have been tested and shown to be otherwise. The materials and supplies used during the method validation process have been tested and shown to be clean. These items are listed below in Section 6.

4.2. To avoid contamination of samples, standards are prepared in a ventilation hood in an area separate from where samples are extracted.

4.3. PTFE products can be a source of PFOA contamination. The use of PTFE in the procedure should be avoided or at least thoroughly tested before use. Polypropylene (PP) or polyethylene (PE, HDPE) products may be used in place of PTFE products to minimize PFOA contamination.

4.3.1. Standards and samples are injected from polypropylene autosampler vials with polypropylene screw caps once. Multiple injections may be performed on Primers when conditioning the instrument for analysis.

4.3.2. Random evaporation losses have been observed with the polypropylene caps causing high IDA recovery after the vial was punctured and sample re-injected. For this reason, it is best to inject standards and samples once in the analytical sequence.

4.3.3. Teflon-lined screw caps have detected PFAS at low concentrations. Repeated injection from the same teflon-lined screw cap have detected PFNA at increasing concentration as each repeated injection was performed, therefore, it is best to use polypropylene screw caps.

4.4. Volumetric glassware and syringes are difficult to clean after being used for solutions containing high levels of PFOA. These items should be labeled for use only with

similarly concentrated solutions or verified clean prior to re-use. To the extent possible, disposable labware is used.

4.5. Both branched and linear PFAS isomers can potentially be found in the environment. Linear and branched isomers are known to exist for PFOS, PFOA, PFHxS, PFBS, EtFOSAA, and MeFOSAA based upon the scientific literature. If multiple isomers are present for one of these PFAS they might be adjacent peaks that completely resolve or not, but usually with a deflection point resolved during peak integration. The later of these peaks matches the retention time of its labeled linear analog. In general, earlier peaks are the branched isomers and are not the result of peak splitting.

As of this writing, only PFOS, PFOA, and PFHxS are commercially available as technical mixtures. These reference standards of the technical mixtures for these specific PFAS are used to ensure that all appropriate peaks are included during peak integration.

4.6. In an attempt to reduce PFOS bias, it is required that *m/z* 499>80 transition be used as the quantitation transition.

4.7. Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. This equates to roughly 0.30 ng/L or 0.015 ug/kg of perfluorohexadecanoic acid expected in all samples and blanks.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Safety Manual, Sacramento Supplement to the CSM, and this document. All work must be stopped in the event of a known or potential compromise to the health or safety of an associate. The situation must be reported **immediately** to a supervisor, the EH&S Staff, or a senior manager.

5.1. Specific Safety Concerns

5.1.1. Preliminary toxicity studies indicate that PFAS could have significant toxic effects. In the interest of keeping exposure levels as low as reasonably achievable, PFAS and PFAS samples must be handled in the laboratory as hazardous and toxic chemicals.

5.1.2. Exercise caution when using syringes with attached filter disc assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

5.1.3. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best

position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.4. Eye protection that satisfies ANSI Z87.1 (as per the TestAmerica Corporate Safety Manual), laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.1.5. Perfluorocarboxylic acids are acids and are not compatible with strong bases.

5.1.6. The use of vacuum systems presents the risk of imploding glassware. All glassware used during vacuum operations must be thoroughly inspected prior to each use. Glass that is chipped, scratched, cracked, rubbed, or marred in any manner must not be used under vacuum. It must be removed from service and replaced.

5.1.7. Glass containers are not to be used for “tumbling” soil samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Acetic Acid (3-2-1)	Corrosive Poison Flammable	10 ppm-TWA 15 ppm-STEL	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.

Material[1]	Hazards	Exposure Limit[2]	Signs and Symptoms of Exposure
Ammonium Hydroxide (3-0-0)	Corrosive Poison	50 ppm-TWA	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage to the upper respiratory tract. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent damage, including blindness. Brief exposure to 5000 PPM can be fatal.
Hexane (2-3-0)	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Hydrochloric Acid (3-0-1)	Corrosive Poison	5 ppm (Ceiling)	Can cause pain and severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause deep ulcerations to skin, permanent eye damage, circulatory failure and swallowing may be fatal.
Methanol (2-3-0)	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Potassium Hydroxide (3-0-1)	Corrosive Poison		Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.
Potassium Persulfate (2-0-1-OX)	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Sodium Hydroxide (3-0-1)	Corrosive Poison	2 mg/cm^3 (Ceiling)	Severe irritant. Can cause severe burns upon inhalation, ingestion, eye or skin contact. Exposure to concentrated solutions may cause severe scarring of tissue, blindness, and may be fatal if swallowed.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6. EQUIPMENT AND SUPPLIES

6.1. 15 mL polypropylene test tubes with polypropylene screw caps.

6.2. 50 mL graduated plastic centrifuge tubes.

6.3. 125 mL HDPE bottles with HDPE screw caps.

6.4. 250 mL HDPE bottles with HDPE screw caps.

6.5. Analytical balance capable of accurately weighing to the nearest 0.0001g, and checked for accuracy each day it is used in accordance with WS-QA-0041.

6.6. Extract concentrator or nitrogen manifold with water bath heating to 50-55°C.

6.7. Syringe filter, Millipore Millex-HV 0.45 um, or equivalent. Do not use PTFE type filters.

6.8. 300 µL autosampler vials, polypropylene, with polypropylene screw caps, Waters PN 1860004112, or equivalent.

6.9. SPE columns

6.9.1. Phenomenex Strata SPE C18, 6 mL, 500 mg, part number 8B-S002-HCH, Waters SepPak C18, 1 to 10g, or equivalent.

6.9.2. Waters Oasis WAX 150 mg/6 cc (PN 186002493) for the cleanup of solids.

6.9.3. Waters Oasis WAX 500 mg/6 cc (PN 186004647) for extraction of PFAS from aqueous sample.

6.9.4. Phenomonex Gemini 3 µm C18 110Å, 50 X 2 mm, Part No. 00B-4439-B0.

6.9.5. Phenomonex Luna 5 µm C18(2) 100Å, 30 X 3 mm, Part No. 00A-4252-Y0.

6.10. Graphitized carbon (Envi-Carb™ or equivalent).

6.11. Vacuum manifold for Solid Phase Extraction (SPE).

6.12. Miscellaneous laboratory apparatus (beakers, test tubes, volumetric flasks, pipettes, etc.). These should be disposable where possible, or marked and segregated for high-level versus low-level use.

6.13. Water bath: Heated with concentric ring cover capable of temperature control (±5°C) up to 95°C. The bath must be used in a fume hood.

6.14. Plastic tub for an ice bath, AKRO-N.S.T. part No. 35-180 or equivalent.

6.15. pH indicator paper, wide range.

6.16. Bottle rotating apparatus for soil extractions.

6.17. Glass fiber filter, Whatman GF/F, catalog number 1825 090 or equivalent.

6.18. Liquid Chromatography/Tandem Mass Spectrometer (LC/MS/MS) – Either of the instruments described below, or equivalent, may be used for this method. Both HPLC are equipped with a refrigerated autosampler, an injection valve, and a pump capable of variable flow rate. The use of a column heater is required to maintain a stable temperature throughout the analytical run. Data is processed using Chrom Peak Review, version 2.1 or equivalent.

6.18.1. SCIEX LC/MS/MS

This system consists of a Shimadzu HPLC interfaced with a SCIEX 5500 Triple Quad MS. The instrument control and data acquisition software is SCIEX Analyst, version 1.6.3 or equivalent.

6.18.1.1. Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.

6.18.1.2. Phenomenex Gemini C_{18} 3 um, 3.0 mm x 100 mm, Part No. 00D-4439-Y0, or equivalent.

6.18.1.3. PFAS Isolator column, Phenomenex Luna C_{18} 5 um, 50 mm x 4.6 mm, part no. 00B-4252-E0 or equivalent. This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.

6.18.2. Waters LC/MS/MS

This consists of a Waters Acquity UPLC system interfaced with a Waters Quattro Premier tandem mass spectrometer. The instrument control and data acquisition software is MassLynx version 4.1, or equivalent.

6.18.2.1. Analytical column: Waters Acquity UPLC BEH C18 1.7 um, 3.0 mm x 150 mm, Part No. 186004690

6.18.2.2. PFAS Isolator column, Waters Acquity UPLC BEH Shield RP-18, 1.7 um, 2.1 mm x 50 mm, PN 186004476, or equivalent. This is plumbed between the UPLC pumps and autosampler valve to minimize PFAS background from the UPLC solvent lines and filters.

6.19. Preventive and routine maintenance is described in the table below

HPLC/MS/MS Preventative Maintenance	
As Needed: Change pump seals. Change in-line filters in autosampler (HPLC). Check/replace in-line frit if excessive pressure or poor performance. Replace column if no change following in-line frit change. Clean corona needle. Replace sample inlet tube in APCI (10.1 cm). Replace fused silica tube in ESI interface. Clean lenses. Clean skimmer. Ballast rough pump 30 minutes. Create all eluents in Reagent module, label eluent containers with TALS label and place 2nd label into maintenance log when put into use.	**Daily (When in use)** Check solvent reservoirs for sufficient level of solvent. Verify that pump is primed, operating pulse free. Check needle wash reservoir for sufficient solvent. Verify capillary heater temperature functioning. Verify vaporizer heater temperature. Verify rough pump oil levels. Verify turbo-pump functioning. Verify nitrogen pressure for auxiliary and sheath gasses. Verify that corona and multiplier are functioning.
Semi-Annually Replace rough-pump oil (4-6 months). Replace oil mist and odor elements. Replace activated alumina filter if applicable	**Annually** Vacuum system components including fans and fan covers. Clean/replace fan filters, if applicable.

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1. Acetic acid, glacial

7.1.2. Ammonium acetate (20 mM in water): Prepared by weighing 1.509g of ammonium acetate and dissolving in 1L of water. The resultant solution is filtered through a 0.22um filter before use. This solution has volatile components, thus it should be replaced every 7 days or sooner.

7.1.3. Ammonium hydroxide (NH4OH), 0.3% in methanol: Prepared by diluting 12mL of ammonium hydroxide into 4L of methanol.

7.1.4. Hexane

7.1.5. Hydrochloric acid (HCl), 2.0 M solution in water

7.1.6. Hydrochloric acid (HCl), concentrated, reagent grade

7.1.7. Methanol

7.1.8. Potassium hydroxide (KOH), 0.4% in methanol: Prepared by weighing 16g of potassium hydroxide and dissolving in 4L of methanol.

7.1.9. Potassium persulfate, reagent grade

7.1.10. Ottawa Sand

7.1.11. Sodium hydroxide (NaOH), 0.1N, in water: Prepared by diluting 400mL of 1N NaOH into 3.6L of water for a total volume of 4L.

7.1.12. Sodium hydroxide (NaOH), 10N, reagent grade

7.1.13. Water, Nanopure or Millipore, must be free of interference and target analytes

7.2. Standards

7.2.1. PFAS are purchased as high purity solids (96% or greater) or as certified solutions. Standard materials are verified compared to a second source material at the time of initial calibration. The solid stock material is stored at room temperature or as specified by the manufacturer or vendor.

7.2.1.1. Per the Certificate of Analysis for labeled perfluorohexadecanoic acid ($13C_2$-PFHxDA) produced by Wellington Laboratories, the stock standard contains roughly 0.3% of native perfluorohexadecanoic acid. This equates to roughly 0.30 ng/L or 0.015 ug/kg of perfluorohexadecanoic acid expected in all samples and blanks.

7.2.2. If solid material is used for preparing a standard, stock standard solutions are prepared from the solids and are stored at $4 \pm 2°C$. Stock standard solutions should be brought to room temperature before using. Standards are monitored for signs of degradation or evaporation. Standard solutions must be replaced at least annually from the date of preparation.

7.2.3. PFBS, PFHxS, PFHpS, PFOS, PFDS, MPFOS, and many other PFAS are not available in the acid form, but rather as their corresponding salts, such as sodium or potassium. The standards are prepared and corrected for their salt content according to the equation below.

$Mass_{acid}$ = Measured $Mass_{salt}$ × MW_{acid} / MW_{salt}

Where: MW_{acid} is the molecular weight of PFAA

MW_{salt} is the molecular weight of the purchased salt.

7.2.4. For example, the molecular weight of PFOS is 500.1295 and the molecular weight of NaPFOS is 523.1193. Therefore, the amount of NaPFOS used must be adjusted by a factor of 0.956.

7.3. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of PFCA and PFSA stock solutions in 80% methanol/water. The calibration stock solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.4. Initial Calibration (ICAL) Levels (ng/mL)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
Perfluoroalkylcarboxylic acids (PFCAs)							
PFBA	0.5	1.0	5.0	20	50	200	400
PFPeA	0.5	1.0	5.0	20	50	200	400
PFHxA	0.5	1.0	5.0	20	50	200	400
PFHpA	0.5	1.0	5.0	20	50	200	400
PFOA	0.5	1.0	5.0	20	50	200	400
PFNA	0.5	1.0	5.0	20	50	200	400
PFDA	0.5	1.0	5.0	20	50	200	400
PFUdA	0.5	1.0	5.0	20	50	200	400
PFDoA	0.5	1.0	5.0	20	50	200	400
PFTrDA	0.5	1.0	5.0	20	50	200	400
PFTeDA	0.5	1.0	5.0	20	50	200	400
PFHxDA	0.5	1.0	5.0	20	50	200	400
PFODA	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonic acids (PFSAs)							
PFBS	0.5	1.0	5.0	20	50	200	400
PFPeS	0.5	1.0	5.0	20	50	200	400
PFHxS *	0.5	1.0	5.0	20	50	200	400
PFHpS	0.5	1.0	5.0	20	50	200	400
PFOS *	0.5	1.0	5.0	20	50	200	400
PFNS	0.5	1.0	5.0	20	50	200	400
PFDS	0.5	1.0	5.0	20	50	200	400
PFDoS	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonamides (FOSA)							

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
FOSA	0.5	1.0	5.0	20	50	200	400
Perfluorinated sulfonamidoacetic acids (FOSAA)							
EtFOSAA	0.5	1.0	5.0	20	50	200	400
MeFOSAA	0.5	1.0	5.0	20	50	200	400
Fluorotelomer sulfonates (FTS)							
4:2 FTS	0.5	1.0	2.0	20	50	200	400
6:2 FTS	0.5	1.0	5.0	20	50	200	400
8:2 FTS	0.5	1.0	5.0	20	50	200	400
10:2 FTS	0.5	1.0	5.0	20	50	200	400
Labeled Isotope Dilution Analytes (IDA)							
13C4-PFBA	50	50	50	50	50	50	50
13C5-PFPeA	50	50	50	50	50	50	50
13C2-PFHxA	50	50	50	50	50	50	50
13C4-PFHpA	50	50	50	50	50	50	50
13C4-PFOA	50	50	50	50	50	50	50
13C5-PFNA	50	50	50	50	50	50	50
13C2-PFDA	50	50	50	50	50	50	50
13C2-PFUdA	50	50	50	50	50	50	50
13C2-PFDoA	50	50	50	50	50	50	50
18O2-PFHxS	50	50	50	50	50	50	50
13C4-PFOS	50	50	50	50	50	50	50
13C3-PFBS	50	50	50	50	50	50	50
13C2-PFTeDA	50	50	50	50	50	50	50
13C2-PFHxDA	50	50	50	50	50	50	50
13C8-FOSA	50	50	50	50	50	50	50
d5-EtFOSAA	50	50	50	50	50	50	50
d3-MeFOSAA	50	50	50	50	50	50	50
M2-4:2FTS ǂ	50	50	50	50	50	50	50
M2-6:2FTS	50	50	50	50	50	50	50
M2-8:2FTS	50	50	50	50	50	50	50
Internal Standard (IS)							
13C2-PFOA	50	50	50	50	50	50	50

* Both branched and linear isomers are used.

ǂ - This compound is used as a reverse surrogate for the TOP analysis.

Note: Sample extracts are in 80% MeOH/H_2O.

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
Fluorinated Replacement Chemicals							
HFPO-DA	0.5	1.0	5.0	20	50	200	400
9CI-PF3ONS	0.5	1.0	5.0	20	50	200	400

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7
Fluorinated Replacement Chemicals							
(F53B major)							
11Cl-PF3OUdS (F53B minor)	0.5	1.0	5.0	20	50	200	400
Dona	0.5	1.0	5.0	20	50	200	400
Labeled Isotope Dilution Analytes							
13C3-HFPO-DA	0.5	1.0	5.0	20	50	200	400

Note: Sample extracts are in 80% MeOH/H_2O.

***Note**: The above calibration limits are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program. The concentration of the calibration solutions for non-concentrated extracts is 1/20th the levels indicated above.*

7.4.1. A technical (qualitative) grade PFOA standard which contains both linear and branched isomers is used as a retention time (RT) marker. This is used to integrate the total response for both linear and branched isomers of PFOA in environmental samples while relying on the initial calibration with the linear isomer quantitative standard This technical (qualitative) grade PFOA standard is analyzed initially, after an initial calibration when a new column is installed or when significant changes are made to the HPLC parameters.

7.5. Initial Calibration Verification Standard (ICV)

A second source solution for PFAS is purchased from the same vendor; the PFC-MXB contains most of the target analytes in this mixture and is used as an ICV. A few compounds are not available in this mixture, may not be available as another lot, and are not available from another vendor. For these analytes only, a second analyst may prepare a second source standard from the same source as the ICAL to produce an ICV. The recommended concentration of the ICV standard should be in the mid-range of the calibration curve. The concentration may be adjusted if the initial calibration levels are changed or altered. The IDA and IS are added at a fixed concentration of 50 ng/mL.

7.6. LCS/Matrix PFC Spike Solution, 20 ng/mL

The PFC spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at a concentration of 20 ng/mL in methanol.

7.7. PFC Isotope Dilution Analyte Solution, 50 ng/mL

The PFC-IDA solution is prepared by diluting all labeled PFAS to produce a solution containing each compound at a concentration of 50 ng/mL in methanol.

7.8. Reverse Surrogate Solution, 1000 ng/mL

The reverse surrogate solution is prepared by diluting M2-4:2 FTS to produce a solution containing this compound at a concentration of 1000 ng/mL in methanol. This is added to all samples for the TOP assay to monitor the efficiency of the oxidation process.

7.9. Internal Standard Solution, 250 ng/mL

The internal standard solution is prepared by diluting 13C2-PFOA to produce a solution containing this compound at a concentration of 250 ng/mL in methanol. This is added to all extracts prior to analysis. The internal standard solution used for the non-concentrated extracts is at a concentration of 50 ng/mL.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Soil samples are collected in pre-cleaned 8 oz. HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6°C for shipment to the laboratory.

8.1.1. Water samples collected from a known chlorinated source should be preserved with Trizma.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6°C. Water samples must be extracted within 14 days of collection. Soil samples must also be extracted within 14 days of collection. Tissue samples must be extracted within 1 year of collection if stored at -20°C. Extracts must be refrigerated at 0 - 6°C, and analyzed within 40 days from extraction.

***Note**: As of this writing, Method 537 provides for a 14 day holding time for water samples preserved with Trizma buffer. The scientific literature indicates that perfluorinated substances are highly persistent in the environment. TestAmerica Sacramento has conducted time stability studies that support a 14 day holding time for aqueous samples with and without Trizma preservation. TestAmerica Denver has conducted stability studies indicating that medium- and low-level solutions of PFOA are stable for at least three months in polystyrene and polypropylene plastics at 0-6°C. The 14/40 day holding times given above are based on the stability study and general EPA convention for the holding time of extractable organic compounds in water and soil.*

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability (IDOC)

The initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.2. Batches are defined at the sample preparation step. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to

analyze prepared extracts on the same instrument or in the same sequence. Refer to the QC program document (WS-PQA-003) for further details of the batch definition.

9.2.1. The quality control batch is a set of up to 20 samples of the same matrix processed using the same procedure and reagents within the same time period. The quality control batch must contain a matrix spike/matrix spike duplicate (MS/MSD), a laboratory control sample (LCS) and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count toward the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD, an LCSD may be substituted if batch precision is required by the program or client. In the event that multiple MS/MSDs are run with a batch due to client requirements, the additional MS/MSDs do not count toward the maximum 20 samples in a batch.

9.3. One method blank (MB, laboratory reagent blank) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. For aqueous samples, the method blank is an aliquot of laboratory reagent water. For solid samples, the method blank is an aliquot of Ottawa sand. The method blank is processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, and then implemented when target analytes are detected in the method blank above the reporting limit or when IDA recoveries are outside of the control limits. Re-extraction of the blank, other batch QC and the affected samples are required when the method blank is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria.

9.3.1. If the MB produces a peak within the retention time window of any of the analytes, determine the source of the contamination and eliminate the interference before processing samples.

9.3.2. The method blank must not contain any analyte at or above the reporting limit, or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.3.3. If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.4. Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.3.5. Refer to WS-PQA-003 for further details of the corrective actions.

9.3.6. Projects performed under the auspices of the DOD/DOE must meet QSM specific criteria for method blanks. Results are acceptable if the blank contamination is less than ½ of the reporting limit/LOQ for each analyte, or less than 1/10 of the regulatory limit, or less than 1/10 of the sample result for the same analyte, whichever is greater. If the method blank does not meet the acceptance criteria, the source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem. Reprepare and reanalyze all field and QC samples associated with the contaminated method blank.

9.4. A laboratory control sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water for aqueous samples and Ottawa sand for solids) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside of the control limits. Re-extraction of the blank, other batch QC, and all associated samples are required if the LCS is deemed unacceptable. See WS-PQA-0003 for specific acceptance criteria. The control limits for the LCS are stored in TALS.

9.5. A matrix spike/matrix spike duplicate (MS/MSD or MS/SD) pair must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. An MS/MSD pair is aliquots of a selected field sample spiked with analytes of known identity and concentration. The MS/MSD pair must be processed in the same manner and at the same time as the associated samples. Spiked analytes with recoveries or precision outside of the control limits must be within the control limits in the LCS. Corrective actions must be documented on a nonconformance memo, and then implemented when recoveries of any spiked analyte are outside of the control limits provided by TALS or by the client.

9.6. A duplicate control sample (LCSD or DCS) may be added when insufficient sample volume is provided to process an MS/MSD pair, or is requested by the client. The LCSD is evaluated in the same manner as the LCS. See WS-PQA-003 for specific acceptance criteria.

9.7. Initial calibration verification (ICV) –A second source standard is analyzed with the initial calibration curve. The concentration should be at the mid range of the curve.

Corrective actions for the ICV include:

- Rerun the ICV.
- Remake or acquire a new ICV.
- Evaluate the instrument conditions.
- Evaluate the initial calibration standards.

- Rerun the initial calibration.

9.8. Isotope Dilution Analytes

9.8.1. The IDA solution is added to each field and QC sample at the time of extraction, as described in Section 11. As described in Section 7, this solution consists of isotopically labeled analogs of the analytes of interest.

9.8.2. IDA recoveries are flagged if they are outside of the acceptance limits (25–150%). Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-nose ratio is greater than 10:1.

9.8.2.1. Evaluate data quality for usability, flag and submit a non-conformance memo for any analytes outside of the recovery criteria, and report if data is deemed not adversely effected.

9.8.2.2. Re-extraction of samples should be performed if the signal-to-noise for any IDA is less than 10:1 or if the IDA recoveries fall below 10%.

9.8.2.2.1. Re-extraction may be necessary under other circumstances when data quality has been determined to be adversely affected.

9.8.2.3. Projects performed under the auspices of the DoD/DOE must meet QSM 5.1 specific criteria for IDA recoveries which are 50-150%. If QC or field samples do not meet these criteria then re-extraction is required.

9.9. Internal Standard

9.9.1. The Internal Standard (IS) is added to each field and QC samples prior to analysis. The CCV IS response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration.

9.9.2. Sample IS response (peak area) must be within ±50% of the response (peak area) in the most recent CCV.

9.9.3. If the IS does not meet criteria, re-analyze the extract. If the IS meets criteria in the second analysis, report that analysis. If the IS does not meet criteria in the second analysis, report the first analysis with narration.

10. CALIBRATION

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-P-003 "Calibration Curves and Selection of Calibration Points".

10.2. Routine instrument operating conditions are listed in the table in Section 11.18.

10.3. Instrument Tuning

Instrument tuning is done initially when the method is first developed and thereafter as needed to maintain the sensitivity and selectivity of the method. Tuning is done by infusing each individual compound (native and IDA) into the mobile phase using a tee fitting at a point just before the entrance to the electrospray probe. The responses for the parent and daughter ions for each compound are observed and optimized for sensitivity and resolution. Mass assignments are reviewed and calibrated if necessary. The mass assignments must be within ± 0.5 amu of the values shown in the table in Section 11.18.

10.3.1. Once the optimal mass assignments (within ±0.5 amu of true) are made immediately following the initial tune, the lowest level standard from the initial calibration curve is assessed to ensure that a signal to noise ratio greater than 10 to 1 (S/N > 10:1) is achieved for each PFAS analyte. The first level standard from the initial calibration curve is used to evaluate the tune stability on an ongoing basis. The instrument mass windows are set initially at ± 0.5 amu of the true value; therefore, continued detection of the analyte transition with S/N > 10:1 serves as verification that the assigned mass remains within ± 0.5 amu of the true value, which meets the DoD/DOE QSM tune criterion. For QSM work, the instrument sensitivity check (section 10.12.4) is also evaluated to ensure that the signal to noise criteria is met.

10.4. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include, but are not limited to, new columns or pump seals. A new calibration is not required after minor maintenance.

10.5. With the exception of the circumstances delineated in policy CA-Q-P-003, it is not acceptable to remove points from a calibration curve. In any event, at least five points must be included in the calibration curve. Average Response Factor and linear fit calibrations require five points, whereas Quadratic (second order) calibrations require six points.

10.6. A fixed injection volume is used for quantitation purposes and is to be the same for both the sample and standards.

10.7. All units used in the calculations must be consistently uniform, such as concentration in ng/mL.

10.8. Initial Calibration

10.8.1. A number of analytical standards of different analyte concentrations are used to generate the curve. Each standard is injected once to obtain the peak response for each analyte at each concentration. These standards define the working range of the analysis.

10.8.1.1. A minimum of five analytical standards is used when using average response factor and/or linear calibration fits.

10.8.1.2. A minimum of six analytical standards is used when a quadratic fit is used to generate the curve.

10.8.2. Calibration is by average response factor, linear fit, or by quadratic fit. Quadratic fit is used for the analyte if the response is non-linear.

10.8.2.1. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated against an identically labeled analog must be < 35% for the curve to be valid.

10.8.2.2. For average response factor (RFa), the relative standard deviation (RSD) for all compounds quantitated against a closely related labeled analog IDA must be < 50% for the curve to be valid.

10.8.2.3. For linear fit, the intercept of the line must be less than ½ the reporting limit, and the coefficient of determination (r2) must be greater than or equal to 0.990 for the curve to be considered valid (or the correlation coefficient (r) > 0.995).

10.8.2.4. The Internal Standard (IS) response (peak area) must not deviate by more than 50% from the average response (peak area) of the initial calibration.

10.8.2.5. Projects performed under the auspices of the DoD/DOE must meet QSM 5.1 specific criteria for initial calibration: The %RSD of the RFS for all analytes must be <20%. Linear or non-linear calibrations must have r^2>0.99 for each analyte. Analytes must be within 70-130% of their true value for each calibration standard.

10.9. Calibration Curve Fits

10.9.1. Linear regression or quadratic curves may be used to fit the data to a calibration function. Detailed descriptions and formulas for each fitting type can be found in SOP CA-Q-P-003, "Calibration Curves and Selection of Calibration Points".

10.9.2. The linear curve uses the following function:

Equation 1

$$y = bx + c$$

Where:

$$y = \frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$$

x = concentration
b = slope
c = intercept

10.9.3. The quadratic curve uses the following function:

Equation 2

$$y = ax^2 + bx + c$$

Where y, x, b, and c are the same as above, and a = curvature.

10.9.4. Evaluation of Calibration Curves

The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- The absolute value of the intercept of a regression line (linear or non-linear) at zero response must be less than the reporting limit.
- There should be no carryover at or above 1/2 MRL after a high CAL standard.

If these criteria are not met, instrument conditions and standards will be checked, and the ICAL successfully repeated before continuing.

10.9.5. Weighting of Calibration Points

In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. Because accuracy at the low end of the curve is very important for this analysis, it is preferable to increase the weighting of the lower concentration points. 1/concentration or 1/x weighting is encouraged. Visual inspection of the line fitted to the data is important in selecting the best fit.

10.10. Initial Calibration Blank (ICB)

10.10.1. Immediately following the ICAL, a calibration blank is analyzed that consists of an injection of 80:20 methanol:water blank containing both IDA and IS.

10.10.2. The result for the calibration blank must be less than the reporting limit.

10.10.3. If the ICB is greater than the reporting limit then the source of contamination must be identified and any necessary cleaning completed, and then the instrument should be recalibrated.

10.10.4. Projects performed under the auspices of the DoD/DOE must meet QSM 5.1 specific criteria for instrument blanks. One is required immediately following the highest standard analyzed and *daily prior to sample analysis*. The instrument blank must be < ½ the LOQ.

10.11. Initial Calibration Verification (ICV)

10.11.1. Following the ICAL and the ICB, an ICV standard obtained from a different source or vendor than the ICAL standards is analyzed. This ICV standard is a mid-range standard.

10.11.2. The recovery for the ICV must meet the appropriate following criteria:

10.11.2.1. The native analyte must be within or equal to 60-140% for all native analytes quantitated against an identically labeled analog IDA.

10.11.2.2. The native analyte must be within or equal to 50-150% for all native analytes quantitated against a closely related labeled analog IDA.

10.11.2.3. The IDA must be within or equal to 50-150%.

10.11.3. Projects performed under the auspices of the DoD/DOE must meet QSM 5.1 specific criteria for the ICV. Analyte concentrations must be within ±30% of their true values for all analytes, IDA and target.

10.11.4. See Section 9.7 for corrective actions in the event that the ICV does not meet the criteria above.

10.12. Continuing Calibration Verification (CCV)

Analyze a CCV at the beginning of a run, the end of a run, and after every 10 samples to determine if the calibration is still valid. The exception is after an acceptable curve and ICV are run 10 samples can be analyzed before a CCV is required. The CCVs are

usually at the mid-level range of the curve and should vary throughout the run from low level (LOQ/RL) to mid level. The curve and ICV do not need to be run every day. To start an analytical run a CCV can be analyzed and if it meets acceptance criteria a run can be started. In addition, the low standard in the curve must be analyzed and must be within ± 50% of the expected value.

10.12.1. The recovery for the CCV standards must be equal to or within 60-140% for all natives quantitated against an identically labeled analog and equal to or within 50% to 150% for all natives quantitated against a closely related labeled analog. The recovery for the IDA must be within or equal to 50-150%.

10.12.2. The Internal Standard (IS) response (peak area) must be within ± 50% from the response (peak area) from the midpoint of the initial calibration.

10.12.2.1. Sample IS response (peak area) must be within ± 50% of the response (peak area) in the most recent CCV.

10.12.3. If this is not achieved, the instrument has drifted outside the calibration limits. The instrument must be recalibrated.

10.12.4. Projects performed under the auspices of the DoD/DOE must meet QSM 5.1 specific criteria for CCV. All analyte concentrations must be within ± 30% of their true value. Additionally, prior to analysis and at least once every 12 hours an instrument sensitivity check (ISC/CCVL) must be analyzed. The analyte concentrations must be at LOQ and the concentrations must be within ± 30% of their true value. This can be used as a CCV.

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an Non-Conformance Memo (NCM). The NCM process is described in more detail in SOP WS-QA-0023. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.2. Water Sample Preparation

11.2.1. Visually inspect samples for the presence of settled and/or suspended sediment/particulates. If present or if the sample is biphasic add IDA prior to any sample decanting or centrifugation. If the sample requires decanting or centrifugation contact the client for guidance prior to such action.

Decanting or filtering of the sample can lead to a low bias.

11.2.2. If authorized by the client to filter the sample, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity or vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration. File an NCM noting the need for filtration.

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.2.3. Weigh the sample container prior to extraction and then weigh the sample container after extraction to determine the initial volume. Unless otherwise directed by client, use the entire sample volume.

11.2.4. Prepare additional aliquots of a field sample for the MS/MSD, if requested.

11.2.5. Prepare two 250 mL aliquots of HPLC-grade water for the method blank and LCS.

11.2.6. Spike the LCS and MS/MSD (if requested) with 0.5 mL of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 40 ng/L.

11.2.7. Add 0.5 mL of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.

11.3. Solid Phase Extraction (SPE) of Aqueous Samples

The automated Zymark Auto-Trace Workstation can be used as long as the program follows these conditions and passes the background check.

11.3.1. Condition the SPE cartridges (Waters WAX, 500 mg/6 cc) by passing the following without drying the column.

Note: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.3.2. Wash with 5.0 mL of 0.3% NH_4OH/methanol.

11.3.3. Wash with 5.0 mL of 0.1N NaOH/water. Close valve when ~ 200 uL remains on top to keep column wet. After this step, the columns cannot go

dry until the completion of loading and rinsing samples.

11.3.4. Appropriately label the columns and add the reservoir to the column.

11.3.5. Add samples to the columns and with vacuum, pull the entire 250 mL aliquot of the sample through the cartridge at a rate of approximately 2 to 5 drops per second.

11.3.6. After the final loading of the sample but before completely passed through the column, rinse the SPE column with 1 mL of water.

11.3.7. After the sample and water rinse have completely passed through the cartridge, allow the column to dry well with vacuum for 15 minutes.

11.4. SPE Column Wash of Aqueous Samples with Hexane

11.4.1. Load the first 5 mL of hexane to soak for five minutes and then elute to waste.

11.4.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

11.4.3. Allow the column to dry with vacuum for 5 to 10 minutes. Columns must be dried before continuing.

11.5. SPE Elution of Aqueous Samples – using 15 mL polypropylene test tubes as receiving tubes in the SPE manifold.

11.5.1. Rinse sample bottles with 5 mL of 0.3% NH_4OH/methanol and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.5.2. Repeat sample bottle to column reservoir rinse and cartridge elution with a second 5 mL aliquot of 0.3% NH_4OH/methanol. The total collection should be approximately 10 mL.

11.5.3. **Note: If the extracts will not be concentrated elute extract with a total of 8 mL of 0.3% NH_4OH/methanol.**

11.5.4. Proceed to Section 11.15.2 (Graphitized Carbon Cleanup) as needed. This required for all DoD/DOE extracts.

11.6. Extract Concentration for Aqueous Extracts (Note, if the extract will not be concentrated, proceed to Section 11.7.)

11.6.1. Prior to concentrating each sample, add 100 uL of water.

11.6.2. Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 100 uL of water remains.

11.6.2.1. This blow down must take a minimum of 3.5 hours.

11.6.2.2. Extracts can not remain in the water bath longer than 5 minutes once concentrated.

11.6.3. Add 300 uL of methanol and mix the contents well using a vortex mixer.

11.6.4. Add 100 uL of Internal Standard (IS) 250 ng/mL concentration solution to each extract and vortex to mix.

11.6.5. This will create an extract with a final solvent composition of 80:20 methanol:water.

11.6.6. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.6.7. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.

11.7. Final volume for non-concentrated extract

11.7.1. If the extract does not undergo concentration add 0.5 mL of IS 50 ng/mL concentration and 2 mL of water to the extract. This will create an extract with a final solvent composition of 80:20 methanol:water.

11.7.1.1. Seal the test tube tightly. Invert container several times and then vortex. Allow extract to settle for 10 minutes prior to moving to the next step.

11.7.2. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.7.3. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps cannot be used due to detection of low level concentration of PFAS.

11.8. Soil, Sediment and Tissue Sample Preparation and Extraction

11.8.1. Visually inspect soil samples for homogeneity.

11.8.1.1. Projects performed under the auspices of the DoD/DOE must have the entire sample homogenized prior to subsampling in accordance with QSM 5.1 criteria (see SOP WS-QA-0018).

11.8.2. Weigh a representative 5 g aliquot of soil, sediment or 1 g of tissue sample into a 50 mL HDPE wide-mouth bottle. Weigh additional sample amounts for the matrix spike and matrix spike duplicate analyses if they are requested.

11.8.3. For the method blank and LCS matrix, use 5 g each of Ottawa sand or 0.1 g of oil.

11.8.4. Spike the LCS and MS/MSD (if requested) with 1.0 mL of the LCS/Matrix PFC Spike solution (Section 7.6). This will result in a sample concentration of 4.0 ng/g.

11.8.4.1. Spike non-concentrated samples at 0.5 mL of LCS/Matrix PFC Spike Solution.

11.8.5. Add 1.0 mL of the IDA PFC solution (Section 7.7) into each sample and QC sample, for a fixed concentration of 50 ng/mL in the final sample vial.

11.8.5.1. Spike non-concentrated samples at 0.5 mL of IDA PFC Solution.

11.8.6. Cap the bottles and allow the spike to settle into the sample matrix. Gently shake the bottles to mix the spike into the matrix.

11.8.7. Add 20 mL of 0.4% KOH/methanol to each sample.

11.8.8. Shake each sample on an orbital shaker at room temperature for 3 hours.

11.8.9. Following the shaking, extract the samples in an ultrasonic water bath for an additional 12 hours.

11.8.10. After the completion of extraction, centrifuge each sample at 3500 rpm for 15 minutes.

11.8.11. Collect and decant the KOH/methanol extract to a new 50 mL centrifuge tube.

11.8.12. Add another 2 mL of 0.4% KOH/methanol solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.

11.8.13. Combine the rinsate to the first corresponding tubes.

11.8.14. To the final KOH/methanol extract, add 2 mL of water to each.

11.8.15. Concentrate the KOH/methanol/water extract under nitrogen to less than 2 mL, and dilute with water to 15 mL final volume.

11.8.16. Acidify with 80 uL of glacial acetic acid, and mix the contents well with vortex mixer. Check the pH to ensure pH is between 6 to 8.

11.8.17. Centrifuge at 3500 rpm for 15 minutes.

11.9. Solid Extract Cleanup by SPE

Set up WAX 150 mg/6 cc SPE columns for sample cleanup using vacuum manifold.

11.9.1. Condition the SPE cartridges by passing the following without drying the column.

Note: *The cartridges should not be allowed to go dry until the final elution step with methanol. At all of the other transition steps, the solvent/sample level should be stopped at the top of the column before the next liquid is added.*

WARNING: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.9.2. Wash with 5.0 mL of 0.3% NH_4OH/methanol.

11.9.3. Wash with 10 mL of 0.1N NaOH/water. Close valve when ~ 500uL remains on top of column to keep column wet. *After this step, the columns cannot go dry until the completion of loading and rinsing samples.*

11.9.4. Add extracts to the columns and with vacuum, pull the entire extracts through the cartridge at rate of approximately 3 to 5 drops per second.

11.9.5. Rinse the sample tube with 5 mL of water and add to the SPE column.

11.9.6. Dry the columns with vacuum for 15 minutes.

11.10. SPE Column Wash of Solid Extracts with Hexane

11.10.1. Load the first 5 mL of hexane to soak for five minutes, and elute to waste.

11.10.2. Load the second 5 mL of hexane and elute to waste (without a soaking period).

11.10.3. Allow the column to dry with vacuum for 10 minutes. Columns must be dried before continuing.

11.11. SPE Elution of Solid Extracts – using 15 mL polypropylene test tube as receiving tube in the SPE manifold.

11.11.1. Rinse extraction bottles with 5 mL of 0.3% NH_4OH/methanol and transfer to the column reservoir onto the cartridge. Allow the solution to soak for 5 minutes and then elute into the 15 mL collection tube.

11.11.2. Repeat extract bottle to column reservoir rinse and cartridge elution with a second 5 mL aliquot of 0.3% NH_4OH/methanol. The total collection should be approximately 10 mL.

11.11.3. **Note: If the extracts will not be concentrated elute extract with a total of 8 mL of 0.3% NH_4OH/methanol.**

11.11.4. Proceed to Section 11.15.2 (Graphitized Carbon Cleanup) as needed. This is required for all DoD/DOE extracts.

11.12. Extract Concentration for Solid Samples (Note, if the extract will not be concentrated, proceed to Section 11.7)

11.12.1. Prior to concentrating each sample, add 200 uL of water.

11.12.2. Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 200 uL of water remains.

11.12.2.1. This blow down must take a minimum of 3.5 hours.

11.12.2.2. Extracts can not remain in the water bath longer than 5 minutes once concentrated.

11.12.2.3. Add 600 uL of methanol and mix the contents well using a vortex mixer.

11.12.2.4. Add 200 uL of Internal Standard (IS) 250 ng/mL concentration solution to each extract and vortex to mix.

11.12.3. Transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution.

11.12.4. Seal the vial with a polypropylene screw cap. *Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.*

11.13. Product/Dispersion Samples

11.13.1. Check the solubility of the material in both methanol and water

11.13.1.1. If the material is soluble in water, dilute 0.5 mL of sample into 250 mL of DI water and proceed to Section 11.3 (follow water

extraction procedures). Fortify sample appropriately with IDA or PFC spike solution, see Section 11.2.

11.13.1.2. If the material is soluble in methanol, dilute 1 g (if solid) or 1 mL (if liquid) of material into 10 mL of methanol (MeOH).

11.13.1.2.1. If the material does not completely dissolve, contact your immediate supervisor.

11.13.2. Take 100 uL of the 10 mL solution and dilute it to 10 mL in MeOH.

11.13.3. Take a 1 mL aliquot of this solution (effective dilution of 1000x (1 mg for solid or 0.001 mL for liquid)) and fortify with 0.5 mL of labeled IDA solution (Section 7.7).

11.13.4. DO NOT PASS EXTRACT THROUGH SPE CARTIRIDGE (omit steps 11.9 – 11.11).

11.13.5. Proceed to Section 11.6 of this SOP for extract concentration.

11.14. TOP (Total Oxidizable Precursor) Assay for Aqueous Samples

11.14.1. Prepare 3-250 mL HDPE containers with HPLC grade water to create the needed QC Samples (MB, LCS/LCSD).

11.14.2. Prepare enough 125 mL HDPE containers as needed for all “Pre” and “Post” samples, including QC. Label each appropriately.

11.14.3. Spike the “Pre” and “Post” MB 125 mL containers with 25 uL of the reverse surrogate solution of M2-4:2 FTS (Section 7.8).

11.14.4. Spike the “Pre” and “Post” LCS/LCSD 125 mL containers with 0.5 mL of the LCS Spike solution (Section 7.6), both regular and “add-on”, and 25 uL of the reverse surrogate solution (Section 7.8).

11.14.5. Remove the methanol solvent from all Post QC sample 125 mL containers (MB and LCS/LCSD) by using N2 evaporation.

11.14.6. Add 2g of potassium persulfate and 1.9 mL of 10 M NaOH to each “Post” sample container.

11.14.7. Subsample 100 mL aliquots of water from each field sample and QC from the 250 mL containers into each of the corresponding 125 mL containers for both the “Pre” and “Post” samples. Spike all “Pre” and “Post” samples with 25uL of the reverse surrogate solution (Section 7.8).

11.14.8. Set aside all “Pre” sample containers.

11.14.9. Cap each “Post” sample container, invert 2-3 times prior to placing container into water bath.

11.14.10. Add 2g of potassium persulfate and 1.9 mL of 10N NaOH to each “Post” sample container.

11.14.11. Heat each “Post” sample container in a water bath (KD) at 85°C for 6 hours.

11.14.12. After digestion for 6 hours, place the “Post” sample containers in an ice bath for 30 minutes.

11.14.13. Adjust the pH of “Post” samples and associated QC aliquots to 7 with concentrated HCl. Use pH paper to determine the pH.

11.14.14. Spike both “Pre” and “Post” samples and their associated QC samples with 0.5 mL of PFC IDA solution (Section 7.7), both regular and add-on.

11.14.15. Use the following SPE procedure for both “Pre” and “Post” samples:

11.14.15.1. Set up WAX 150 mg/6 cc SPE columns for sample extraction using a vacuum manifold.

11.14.15.2. Establish a sample loading flow rate of 1 mL/minute for each port of the vacuum manifold, for as many ports as will be used simultaneously during sample loading.

11.14.15.3. Wash/condition the SPE column with 5 mL of 0.3% NH_4OH/Methanol, then 5 mL water.

11.14.15.4. Load 100 mL of sample onto the SPE cartridge at a flow rate of 1 mL/minute.

11.14.15.5. Add 5 mL rinse water

11.14.15.6. After the sample and water rinse have completely passed through the column, allow it to dry well using vacuum with a flow rate of 1 mL/minute for 15 minutes.

11.14.15.7. Wash the SPE column with 10 mL hexane rinse eluting all to waste.

11.14.15.8. Allow the column to dry well using vacuum with a flow rate of 1 mL/minute for 5 minutes. Columns must be dry before continuing.

11.14.15.9. Elute the samples into 15 mL polypropylene test tubes in the SPE manifold by rinsing each 125 mL sample container with 5 mL of 0.3% NH_4OH/methanol, and add to the SPE cartridge as eluent.

11.14.15.10. Repeat with another 5 mL of 0.3% NH_4OH/methanol.

11.14.15.11. Collect the 10 mL of eluent and concentrate per Section 11.6.

11.15. TOP (Total Oxidizable Precursor) Assay for Soil Samples

11.15.1. Weigh representative 2 g aliquots of soil for each "Pre" and "Post" sample into a 50 mL centrifuge tube.

11.15.2. For the method blank and LCS matrix, use 2 g each of Ottawa sand for each "Pre" and "Post" QC sample.

11.15.3. Add 20 mL of 0.4% KOH/methanol to each sample.

11.15.4. Shake each sample on an orbital shaker at room temperature for 3 hours.

11.15.5. Following the shaking, extract the samples in an ultrasonic water bath for an additional 12 hours.

11.15.6. After the completion of extraction, centrifuge each sample at 3500 rpm for 15 minutes.

11.15.7. Collect and decant the KOH/methanol extract to a new 50 mL centrifuge tube.

11.15.8. Add another 2 mL of 0.4% KOH/methanol solution to the residue, briefly shake to mix and centrifuge at 3500 rpm for 15 minutes.

11.15.9. Combine the rinsate to the first corresponding tubes.

11.15.10. Proceed to Section 11.16.2 (Envi-carb clean up)

11.15.11. To the final KOH/methanol extract, add 0.5 mL of water to each.

11.15.12. Concentrate the KOH/methanol/water extract under nitrogen to less than 0.25 mL.

11.15.13. Dilute extract up to 50 mL with water in the centrifuge tube and vortex.

11.15.14. Prepare enough 125 mL HDPE containers as needed for all "Pre" and "Post" samples, including QC. Label each appropriately.

11.15.15. Spike the “Pre” and “Post” MB 125 mL containers with 25 uL of the reverse surrogate solution of M2-4:2 FTS (Section 7.8).

11.15.16. Spike the “Pre” and “Post” LCS/LCSD 125 mL containers with 0.5 mL of the LCS Spike solution and 25 uL of the reverse surrogate solution (Section 7.8).

11.15.17. Remove the methanol solvent from all “Post” QC sample 125 mL containers (MB and LCS/LCSD) by using N2 evaporation.

11.15.18. Add 2g of potassium persulfate and 1.9 mL of 10N NaOH to each “Post” sample container.

11.15.19. Transfer extract from the centrifuge tube to the appropriate 125 mL container.

11.15.20. Rinse the centrifuge container with an additional 50 mL of water and transfer to the appropriate 125 mL container.

11.15.21. Set aside all “Pre” sample containers.

11.15.22. Cap each “Post” sample container, invert 2-3 times prior to placing container into water bath.

11.15.23. Heat each “Post” sample container in a water bath (KD) at 85°C for 6 hours.

11.15.24. After digestion for 6 hours, place the “Post” sample containers in an ice bath for 30 minutes.

11.15.25. Adjust the pH of “Post” samples and associated QC aliquots to 7 with concentrated HCl. Use pH paper to determine the pH.

11.15.26. Spike both “Pre” and “Post” samples and their associated QC samples with 0.5 mL of PFC IDA solution (Section 7.7).

11.15.27. Use the following SPE procedure for both “Pre” and “Post” samples:

11.15.27.1. Set up WAX 150 mg/6 cc SPE columns for sample extraction using a vacuum manifold.

11.15.27.2. Establish a sample loading flow rate of 1 mL/minute for each port of the vacuum manifold, for as many ports as will be used simultaneously during sample loading.

11.15.27.3. Wash/condition the SPE column with 5 mL of 0.3% NH_4OH/Methanol, then 5 mL water.

11.15.27.4. Load 100 mL of sample onto the SPE cartridge at a flow rate of 1 mL/minute.

11.15.27.5. Add 5 mL rinse water

11.15.27.6. After the sample and water rinse have completely passed through the column, allow it to dry well using vacuum with a flow rate of 1 mL/minute for 15 minutes.

11.15.27.7. Wash the SPE column with 10 mL hexane rinse eluting all to waste.

11.15.27.8. Allow the column to dry well using vacuum with a flow rate of 1 mL/minute for 5 minutes. Columns must be dry before continuing.

11.15.27.9. Elute the samples into 15 mL polypropylene test tubes in the SPE manifold by rinsing each 125 mL sample container with 5 mL of 0.3% NH_4OH/methanol, and add to the SPE cartridge as eluent.

11.15.27.10. Repeat with another 5 mL of 0.3% NH_4OH/methanol.

11.15.27.11. Collect the 10 mL of eluent and concentrate per Section 11.6.

11.15.27.12. Note: If the extracts will not be concentrated elute extract with a total of 8 mL (2 4 mL rinses) of 0.3% NH_4OH/methanol.

11.16. Other Types of Sample Cleanup

11.16.1. Freezing technique to remove lipids.

If samples contain lipids then freeze the methanolic extract and QC extracts at -20^{o}C for at least 1 hour. Collect the solvent layer.

11.16.2. Cleanup with graphitized carbon will be applied to all samples as needed but is required for all DoD/DOE extracts.

11.16.2.1. Add 100 mg of graphitized carbon to each sample extract and QC extracts.

11.16.2.2. Shake vigorously and then let sit for 10 minutes.

11.16.2.3. Centrifuge each sample for 2 minutes at 1000 rpm.

11.16.2.4. Decant the solvent layer

11.16.2.5. Proceed to Section 11.6, 11.7 or 11.12 as applicable.

11.17. AFFF Sample Preparation

11.17.1. QC for AFFF samples consists of a method blank, a laboratory control sample and a sample or matrix duplicate only. No matrix spike or matrix spike duplicate is needed.

11.17.2. Perform a 1,000,000 X serial dilution of the AFFF sample. Dilute 1 mL of AFFF sample to 1L with laboratory supplied water. Then dilute 1mL of this dilution to 1L with laboratory supplied water.

11.17.2.1. Be sure to retain all dilutions should the initial analysis warrant re-analysis at higher concentration.

11.17.3. Subsample 2.0 mL of this dilution and fortify with 0.5 mL IDA solution and 0.5mL of IS (50 ng/mL) solution: then add 7.0 mL of methanol.

11.17.4. Transfer a portion of the sample to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the sample for re-injection or dilution.

11.18. Instrument Analysis

Suggested operating conditions are listed in Tables 1-7 for the Waters and SCIEX LCMS systems:

Table 1 - Recommended Instrument Operating Conditions

HPLC Conditions (Shimadzu HPLC)				
Column (Column temp = 45°C)	Phenomenex Gemini 3 µm C18 110Å, 50 X 2 mm			
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol			
Gradient Program	**Time**	**%A**	**%B**	**Flow Rate - mL/min**
	0	90	10	0.60
	0.1	45	55	0.60
	4.5	1	99	0.60
	4.95	1	99	0.60
	5	90	10	0.60
	Maximum pressure limit = 5,000 psi			
Injection Size	2 µL (fixed amount throughout the sequence). If non-concentrated extract then use 20 uL.			
Run Time	~6.6 minutes			
Mass Spectrometer Interface Settings (SCIEX 5500)				
MS Interface Mode	ESI Negative Ion. Minimum of 10 scans/peak.			
Ion Spray Voltage (kV)	4.5			

Table 1 - Recommended Instrument Operating Conditions	
HPLC Conditions (Shimadzu HPLC)	
Entrance Potential (V)	5
Declustering Potential (V)	25
Desolvation Temp	600ºC
Curtain Gas	35 psi
Collision Gas	8 psi

Table 2 - Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings (SCIEX 5500)								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
PFBA	Native analyte	212.9 > 169	0.011	-5	-12	-25	-31	1.74
13C4-PFBA	IDA	217 > 172	0.011	-5	-12	-25	-31	1.74
PFBS	Native analyte	298.9 > 80	0.011	-6	-58	-55	-37	1.76
PFBS_2	Native analyte	298.9 > 99	0.011	-5	-40	-55	-12	1.76
13C3-PFBS	IDA	301.9 > 83	0.011	-5	-40	-55	-12	1.76
PFPeA	Native analyte	262.9 > 219	0.011	-7	-12	-20	-34	1.99
13C5-PFPeA	IDA	267.9 > 223	0.011	-7	-12	-20	-35	1.99
4:2 FTS	Native analyte	327 > 307	0.011	-7	-32	-50	-10	2.06
M2-4:2FTS	Reverse Surrogate	329 > 81	0.011	-7	-32	-50	-10	2.06
PFHxA	Native analyte	313 > 269	0.011	-5	-12	-25	-37	2.25
PFHxA_2	Native analyte	313 > 119	0.011	-5	-12	-25	-37	2.25
13C2-PFHxA	IDA	315 > 270	0.011	-5	-12	-25	-38	2.25
PFHpA	Native analyte	363 > 319	0.011	-6	-12	-25	-41	2.57
PFHpA_2	Native analyte	363 > 169	0.011	-6	-12	-25	-41	2.57
13C4-PFHpA	IDA	367 > 322	0.011	-6	-12	-25	-41	2.57
PFPeS	Native analyte	349 > 80	0.011	-9	-66	-57	-40	2.15
PFPeS_2	Native analyte	349 > 99	0.011	-9	-40	-57	-12	2.15
PFHxS	Native analyte	399 > 80	0.011	-12	-74	-60	-43	2.59
PFHxS_2	Native analyte	399 > 99	0.011	-12	-74	-60	-43	2.59
18O2-PFHxS	IDA	403 > 84	0.011	-12	-74	-60	-43	2.59
6:2 FTS	Native analyte	427 > 407	0.011	-7	-32	-50	-10	2.91
M2-6:2FTS	IDA	429 > 81	0.011	-7	-32	-50	-10	2.91
PFOA	Native analyte	413 > 369	0.011	-6	-14	-25	-44	2.93
PFOA_2	Native analyte	413 > 169	0.011	-5	-22	-25	-12	2.93
13C4-PFOA	IDA	417 > 372	0.011	-6	-14	-25	-44	2.93
13C2-PFOA	IS	415 > 370	0.011	-6	-14	-25	-44	2.93
PFHpS	Native analyte	449 > 80	0.011	-11	-88	-65	-46	2.94
PFHpS_2	Native analyte	449 > 99	0.011	-11	-88	-65	-46	2.94
PFNA	Native analyte	463 > 419	0.011	-6	-14	-25	-47	3.29
PFNA_2	Native analyte	463 > 169	0.011	-6	-14	-25	-47	3.29
13C5-PFNA	IDA	468 > 423	0.011	-6	-14	-25	-48	3.29

Table 2 - Recommended Instrument Operating Conditions								
Mass Spectrometer Scan Settings (SCIEX 5500)								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
PFOS	Native analyte	499 > 80	0.011	-9	-108	-65	-50	3.29
PFOS_2	Native analyte	499 > 99	0.011	-5	-58	-65	-12	3.29
PFNS	Native analyte	549 > 80	0.011	-10	-113	-75	-52	3.40
PFNS_2	Native analyte	549 > 99	0.011	-8	-71	-75	-12	3.40
PFDoS	Native analyte	699 > 80	0.011	-11	-76	-10	-11	4.48
PFDoS_2	Native analyte	699 >99	0.011	-11	-130	-10	-5	4.48
13C4-PFOS	IDA	503 > 80	0.011	-9	-108	-65	-50	3.29
PFDA	Native analyte	513 > 469	0.011	-6	-16	-25	-51	3.65
PFDA_2	Native analyte	513 > 169	0.011	-6	-16	-25	-51	3.65
13C2-PFDA	IDA	515 > 470	0.011	-6	-16	-25	-51	3.65
8:2 FTS	Native analyte	527 > 507	0.011	-7	-40	-50	-15	3.65
10:2 FTS	Native analyte	627 > 607	0.011	-7	-38	-110	-5	4.25
M2-8:2FTS	IDA	529 > 81	0.011	-7	-40	-50	-15	3.65
PFOSA	Native analyte	498 > 78	0.011	-8	-85	-60	-50	3.7
13C8-PFOSA	IDA	506 > 78	0.011	-8	-85	-60	-50	3.7
N-MeFOSAA	Native analyte	570 > 419	0.011	-7	-36	-40	-15	3.82
d3-MeFOSAA	IDA	573 > 419	0.011	-7	-36	-40	-15	3.82
PFDS	Native analyte	599 > 80	0.011	-11	-118	-85	-54	3.96
PFDS_2	Native analyte	599 > 99	0.011	-11	-118	-85	-54	3.96
PFUdA	Native analyte	563 > 519	0.011	-7	-18	-25	-54	3.97
PFUdA_2	Native analyte	563 > 169	0.011	-7	-18	-25	-54	3.97
13C2-PFUdA	IDA	565 > 520	0.011	-7	-18	-25	-54	3.97
N-EtFOSAA	Native analyte	584 > 419	0.011	-7	-36	-50	-15	3.99
d5-EtFOSAA	IDA	589 > 419	0.011	-7	-36	-50	-15	3.99
PFDoA	Native analyte	613 > 569	0.011	-5	-18	-25	-54	4.3
PFDoA_2	Native analyte	613 > 169	0.011	-5	-18	-25	-54	4.3
13C2-PFDoA	IDA	615 > 570	0.011	-5	-18	-25	-54	4.3
PFTrDA	Native analyte	663 > 619	0.011	-7	-20	-25	-54	4.56
PFTrDA_2	Native analyte	663 > 169	0.011	-7	-20	-25	-54	4.56
PFTeDA	Native analyte	713 > 169	0.011	-2	-22	-25	-10	4.79
PFTeDA_2	Native analyte	713 > 219	0.011	-7	-36	-25	-30	4.79
13C2-PFTeDA	IDA	715 > 670	0.011	-2	-22	-25	-10	4.79
PFHxDA	Native analyte	813 > 769	0.011	-7	-24	-25	-54	5.25
PFHxDA_2	Native analyte	813 > 169	0.011	-7	-24	-25	-54	5.25
13C2-PFHxDA	IDA	815 > 770	0.011	-7	-24	-25	-54	5.25
PFODA	Native analyte	913 > 869	0.011	-7	-26	-25	-54	5.55
PFODA_2	Native analyte	913 > 169	0.011	-7	-26	-25	-54	5.55

Table 3 - Recommended Instrument Operating Conditions Mass Spectrometer Scan Settings (SCIEX 5500) for Fluorinated Replacement Chemicals								
Compound	Comments	Reaction (MRM)	Dwell (sec)	Ent. Pot. (V)	Col. Energy (V)	Declu. Pot. (V)	Cell Exit Pot. (V)	Typ RT (Min)
HFPO-DA	Native analyte	329.1 > 285	0.011	-10	-6	-48	-17	2.06
13C3-HFPO-DA	IDA	332.1 > 287	0.011	-10	-10	-40	-17	2.06
9Cl-PF3ONS (F53B major)	Native analyte	531 > 351	0.011	-10	-30	-120	-17	3.23
11Cl-PF3OUdS (F53B minor)	Native analyte	631 > 451	0.011	-10	-40	-160	-17	3.84
Dona	Native analyte	377 > 251	0.011	-10	-16	-55	-17	2.33
Dona_2	Native analyte	377 > 85	0.011	-10	-35	-55	-17	2.33

Table 4 - Retention Times & Quantitation (SCIEX 5500)				
Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBA	1.54	13C4-PFBA	1.54	Isotope Dilution
PFPeA	1.56	13C5-PFPeA	1.56	Isotope Dilution
PFBS	1.78	13C3-PFBS	1.78	Isotope Dilution
PFHxA	2.03	13C2-PFHxA	2.03	Isotope Dilution
PFPeS	2.06	13C3-PFBS	1.78	Isotope Dilution
PFHpA	2.36	13C4-PFHpA	2.36	Isotope Dilution
PFHxS	2.37	18O2-PFHxS	2.37	Isotope Dilution
PFOA	2.71	13C4-PFOA	2.71	Isotope Dilution
PFHpS	2.72	13C4-PFOS	3.09	Isotope Dilution
PFNA	3.09	13C5-PFNA	3.09	Isotope Dilution
PFOS	3.09	13C4-PFOS	3.09	Isotope Dilution
PFNS	3.40	13C4-PFOS	3.09	Isotope Dilution
PFDA	3.45	13C2-PFDA	3.45	Isotope Dilution
FOSA	3.43	13C8-FOSA	3.43	Isotope Dilution
PFDS	3.77	13C4-PFOS	3.09	Isotope Dilution
PFUdA	3.78	13C2-PFUdA	3.78	Isotope Dilution
PFDoA	4.07	13C2-PFDoA	4.07	Isotope Dilution
PFTrDA	4.34	13C2-PFDoA	4.07	Isotope Dilution
PFDoS	4.48	13C4-PFOS	3.09	Isotope Dilution
PFTeDA	4.58	13C2-PFTeDA	4.58	Isotope Dilution
PFHxDA	4.99	13C2-PFHxDA	4.99	Isotope Dilution
PFODA	5.34	13C2-PFHxDA	4.99	Isotope Dilution
EtFOSAA	3.78	d5-EtFOSAA	3.78	Isotope Dilution
MeFOSAA	3.61	d3-MeFOSAA	3.60	Isotope Dilution
4:2 FTS	1.98	13C3-PFBS	1.78	Isotope Dilution

Table 4 - Retention Times & Quantitation (SCIEX 5500)				
Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
6:2FTS	2.69	M2-6:2FTS	2.69	Isotope Dilution
8:2FTS	3.44	M2-8:2FTS	3.44	Isotope Dilution
HFPO-DA	2.06	13C3-HFPO-DA	2.06	Isotope Dilution
9Cl-PF3ONS (F53B major)	3.23	13C4-PFOS	3.09	Isotope Dilution
11Cl-PF3OUdS (F53B minor)	3.84	13C4-PFOS	3.09	Isotope Dilution
Dona	2.33	13C4-PFOS	3.09	Isotope Dilution
10:2 FTS	4.25	M2-8:2 FTS	3.44	Isotope Dilution

Table 5 - Recommended Instrument Operating Conditions						
HPLC Conditions (Waters Acquity UPLC)						
Column (Column temp = 50°C)	Waters Acquity BEH 1.7µm C18, 3.0 x 150 mm					
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol					
Gradient Program	**Time**	**%A**	**%B**	**Curve**	**Flow Rate - mL/min.**	
	0	98	2	6	0.30	
	1	98	2	6	0.30	
	2	50	50	6	0.30	
	12	10	90	6	0.30	
	12.5	0	100	6	0.30	
	16	0	100	6	0.30	
	16.2	98	2	6	0.30	
	Maximum pressure limit = 15,000 psi					
Injection Size	10 µL (fixed amount throughout the sequence)					
Run Time	~20 minutes					
Mass Spectrometer Interface Settings (Quattro Premier XE)						
MS Interface Mode	ESI Negative Ion. Minimum of 10 scans/peak.					
Capillary (kV)	2.8					
Cone (V)	Varies from 8.0 to 65					
Extractor (V)	3					
Source Temp	135ºC					
Desolvation Temp	350ºC					
Cone Gas (nitrogen) Flow	25 L/hour					
Desolvation Gas (nitrogen) Flow	1100 L/hour					

Table 6 - Recommended Instrument Operating Conditions						
Mass Spectrometer Scan Settings (Quattro Premier XE)						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
PFBA	Native analyte	213 > 169	0.02	8	10	1
13C4-PFBA	IDA	217 > 172	0.02	12	10	1
PFPeA	Native analyte	263 > 219	0.02	10	10	2
13C5-PFPeA	IDA	268 > 223	0.02	11	9	2
PFBS	Native analyte	299 > 80	0.02	45	35	2
PFBS_2	Native analyte	299 > 99	0.02	45	35	2
13C3-PFBS	IDA	302 > 83	0.02	45	35	2
PFHxA	Native analyte	313 > 269	0.02	10	10	3
PFHxA_2	Native analyte	313 > 119	0.02	10	10	3
13C2-PFHxA	IDA	315 > 270	0.02	12	9	3
PFHpA	Native analyte	363 > 319	0.02	10	10	4
PFHpA_2	Native analyte	363 > 169	0.02	10	10	4
13C4-PFHpA	IDA	367 > 322	0.02	12	10	4
PFHxS	Native analyte	399 > 80	0.02	55	35	4
PFHxS_2	Native analyte	339 > 99	0.02	55	35	4
18O2-PFHxS	IDA	403 > 84	0.02	50	40	4
PFOA	Native analyte	413 > 369	0.02	12	10	5
PFOA_2	Native analyte	413 > 169	0.02	12	10	5
13C2-PFOA	IS	415 > 370	0.02	12	12	5
13C4-PFOA	IDA	417 > 372	0.02	12	12	5
PFHpS	Native analyte	449 > 80	0.02	60	38	5
PFHpS_2	Native analyte	449 > 99	0.02	60	38	5
PFNA	Native analyte	463 > 419	0.02	16	10	7
PFNA_2	Native analyte	463 > 169	0.02	16	10	7
13C5-PFNA	IDA	468 > 423	0.02	12	12	7
PFOS	Native analyte	499 > 80	0.02	60	40	6
PFOS_2	Native analyte	499 > 99	0.02	60	40	6
PFNS	Native analyte	549 > 80	0.02	60	40	6
PFNS_2	Native analyte	549 > 99	0.02	60	40	6
13C4-PFOS	IDA	503 > 80	0.02	35	48	6
PFDA	Native analyte	513 > 469	0.02	16	12	8
PFDA_2	Native analyte	513 > 169	0.02	16	12	8
13C2-PFDA	IDA	515 > 470	0.02	14	12	8
PFUdA	Native analyte	563 > 519	0.02	15	12	10
PFUdA_2	Native analyte	563 > 169	0.02	15	12	10
13C2-PFUdA	IDA	565 > 520	0.02	14	12	10
PFDS	Native analyte	599 > 80	0.02	74	48	10
PFDS_2	Native analyte	559 > 99	0.02	74	48	10
FOSA	Native analyte	498 > 78	0.02	40	32	9

Table 6 - Recommended Instrument Operating Conditions						
Mass Spectrometer Scan Settings (Quattro Premier XE)						
Compound	Comments	Reaction (MRM)	Dwell (sec)	Cone Volt.	Col. Energy	Function Number
13C8-FOSA	IDA	506 > 78	0.02	48	32	9
PFDoA	Native analyte	613 > 569	0.02	15	14	11
PFDoA_2	Native analyte	613 > 169	0.02	15	14	11
13C2-PFDoA	IDA	615 > 570	0.02	16	12	11
PFTrDA	Native analyte	663 > 619	0.02	12	12	11
PFTrDA_2	Native analyte	663 > 169	0.02	12	12	11
PFTeDA	Native analyte	713 > 169	0.02	12	18	11
PFTeDA_2	Native analyte	713 > 219	0.02	12	18	11
13C2-PFTeDA	IDA	715 > 670	0.02	15	15	11
PFHxDA	Native analyte	813 > 769	0.02	18	15	12
PFHxDA_2	Native analyte	813 > 169	0.02	18	15	12
PFODA	Native analyte	913 > 869	0.02	20	16	12
PFODA_2	Native analyte	913 > 169	0.02	20	16	12
13C2-PFHxDA	IDA	815 > 770	0.02	18	15	12
EtFOSAA	Native analyte	584 > 419	0.02	35	20	9
d5-EtFOSAA	IDA	589 > 419	0.02	30	25	9
MeFOSAA	Native analyte	570 > 419	0.02	30	28	9
d3-MeFOSAA	IDA	573 > 419	0.02	30	25	9
4:2FTS	Native analyte	327 > 307	0.02	40	30	5
M2-4:2FTS	Reverse Surrogate	329 > 81	0.02	40	30	5
6:2FTS	Native analyte	427 > 407	0.02	40	30	5
M2-6:2FTS	IDA	429 > 81	0.02	40	28	5
8:2FTS	Native analyte	527 > 507	0.02	40	28	8
M2-8:2FTS	IDA	529 > 81	0.02	40	28	8

Table 7 - Recommended Instrument Operating Conditions				
Retention Times & Quantitation (Quattro Premier XE)				
Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBA	4.77	13C4-PFBA	4.79	Isotope Dilution
PFPeA	5.90	13C5-PFPeA	5.92	Isotope Dilution
PFBS	6.01	13C3-PFBS	6.01	Isotope Dilution
PFHxA	7.22	13C2-PFHxA	7.25	Isotope Dilution
PFPeS	7.20	18O2-PFHxS	8.64	Isotope Dilution
PFHpA	8.57	13C4-PFHpA	8.59	Isotope Dilution
PFHxS	8.60	18O2-PFHxS	8.64	Isotope Dilution
PFOA	9.80	13C4-PFOA	9.83	Isotope Dilution
PFHpS	9.80	13C4-PFOS	10.90	Isotope Dilution
PFNA	10.88	13C5-PFNA	10.92	Isotope Dilution

Table 7 - Recommended Instrument Operating Conditions				
Retention Times & Quantitation (Quattro Premier XE)				
Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFOS	10.87	13C4-PFOS	10.90	Isotope Dilution
PFNS	11.70	13C4-PFOS	10.90	Isotope Dilution
PFDA	11.82	13C2-PFDA	11.86	Isotope Dilution
FOSA	12.41	13C8-FOSA	12.46	Isotope Dilution
PFDS	12.57	13C4-PFOS	10.90	Isotope Dilution
PFUdA	12.62	13C2-PFUdA	12.66	Isotope Dilution
PFDoA	13.32	13C2-PFDoA	13.34	Isotope Dilution
PFTrDA	13.91	13C2-PFDoA	13.34	Isotope Dilution
PFTeDA	14.39	13C2-PFTeDA	14.39	Isotope Dilution
PFHxDA	15.16	13C2-PFHxDA	15.16	Isotope Dilution
PFODA	15.57	13C2-PFHxDA	15.16	Isotope Dilution
EtFOSAA	12.63	d5-EtFOSAA	12.62	Isotope Dilution
MeFOSAA	12.3	d3-MeFOSAA	12.28	Isotope Dilution
4:2FTS	7.02	13C3-PFBS	6.01	Isotope Dilution
6:2FTS	10.08	M2-6:2FTS	10.08	Isotope Dilution
8:2FTS	11.95	M2-8:2FTS	11.95	Isotope Dilution

11.18.1. Post Spike Sample Analysis for AFFF samples

11.18.1.1. This section only applies to aqueous samples prepared by serial dilution instead of SPE that have reported value of <LOQ (RL) for any analyte.

11.18.1.2. Spike aliquots of the sample at the final dilution reported for the sample with all analytes that have reported of <LOQ in the final dilution. The spike must be at the LOQ concentration to be reported with the sample (the < LOQ value).

11.18.1.3. When analyte concentrations are calculated as <LOQ, the spike must recover within 70-130% of its true value.

11.18.1.4. It the recovery does not meet this criteria, the sample, sample duplicate and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.

11.18.2. Tune and calibrate the instrument as described in Section 10.

11.18.3. A typical run sequence is as follows:

- Rinse Blank (RB, not linked to anything)
- Start ICAL with CCVL but called IC in TALS (starts the 12 hour clock or time 0:00)

- Rest of ICAL
- ICB: link to midpoint of ICAL and samples
- ICV: link to midpoint of ICAL and samples (If ICAL good)
- CCB: link to midpoint of ICAL and samples
- PFOA RT marker (as needed)
- Rinse Blank (RB, not linked to anything)
- 10 samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- 10 more samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- Etc.
- CCVL (within 12 hours from CCVL in ICAL, can be the ending CCV and starts 12 hours all over again): if this occurs link to the midpoint of the ICAL/toggle it as opening/closing CCV.
- CCV: link to midpoint of ICAL
- 10 samples: link to midpoint of ICAL
- CCV: link to midpoint of ICAL
- If no ICAL run that day
- CCB: link to CCVIS
- CCVL (starts 12 hour clock): link to CCVIS
- CCVIS: link to midpoint of ICAL
- 10 samples: link to CCVIS
- CCV: link to CCVIS
- 10 samples: link to CCVIS
- CCV: link to CCVIS
- Etc.
- If going over 12 hours in the sequence : CCVL (within 12 hours from CCVL at item 2 above, can be the ending CCV and starts 12 hours all over again): if this occurs link to the CCVIS /toggle as opening and closing CCV.
- CCV: link to CCVIS
- 10 samples: link to CCVIS
- CCV: link to CCVIS

12. CALCULATIONS

12.1. If the concentration of the analyte ions exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. It may be necessary to dilute samples due to matrix.

12.2. Qualitative Identification

12.2.1. The retention times of PFAS with labeled standards should be the same as that of the labeled IDA's to within 0.05 min. For PFAS with no labeled standards, the RT must be within ± 0.3 minutes of the ICV and CCV standards. *Note: The IDA RT and native RT may be offset by 0.02 to 0.04 minutes.*

12.3. The ICAL established in Section 10 is used to calculate concentrations for the extracts.

12.4. Extract concentrations are calculated as below. The first equation applies to the linear fit, the second to the quadratic line fit.

Equation 3

$$\text{Concentration, ng/mL} = \frac{y - c}{b}$$

Equation 4

$$\text{Concentration, ng/mL} = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

Where:

y = $\frac{\text{Area (analyte)}}{\text{Area (IS)}} \times \text{Concentration (IS)}$
x = concentration
a = curvature
b = slope
c = intercept

12.5. Water Sample Result Calculation:

Equation 5

$$\text{Concentration, ng/L} = \frac{C_{ex} V_t}{V_o}$$

Where:

C_{ex} = Concentration measured in sample extract (ng/mL)
V_t = Volume of total extract (mL)
V_o = Volume of water extracted (L)

12.6. Soil Sample Result Calculation:

Equation 6

$$\text{Concentration, } ng/g = \frac{C_{ex} V_t}{W_s D}$$

Where ng/g = µg/kg and:

C_{ex}	=	Concentration measured in sample extract (ng/mL)
V_t	=	Volume of total extract (mL)
W_s	=	Weight of sample extracted (g)
D	=	Fraction of dry solids, which is calculated as follows:

$$\frac{100 - \%\ \text{moisture in sample}}{100} \quad \text{(for dry weight result)}$$

12.7. IDA Recovery Calculation:

Equation 7

$$\%\,\text{Recovery} = \frac{A_t Q_{is}}{A_{is} Q_t RRF_{IDA}} X 100$$

Where ng/g = µg/kg and:

RF_{IDA}	=	Response Factor for IDA compound
A_t	=	Area response for IDA compound
A_{IS}	=	Area Response for IS compound
Q_{IS}	=	Amount of IS added
Q_t	=	Amount of IDA added

12.8. Raw data, calibration summaries, QC data, and sample results are reviewed by the analyst. These must also be reviewed thoroughly by a second qualified person. See the Data Review Policy (WS-PQA-0012). These reviews are documented on the Data Review Checklist.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006 and policy WS-PQA-003. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration of Capability (IDOC)

Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. IDOCs are approved by the

Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

13.4. The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in WS-QA-0006 and policy WS-PQA-003.

14. POLLUTION PREVENTION

14.1. All waste will be disposed of in accordance with Federal, State and Local regulations.

14.2. Solid phase extraction used for water samples greatly reduces the amount of solvent used compared to liquid-liquid extraction.

14.3. Standards and reagents are purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.4. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.5. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless waste is being transferred.

14.6. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

The following waste streams are produced when this method is carried out:

15.1. Assorted test tubes, autovials, syringes, filter discs and cartridges. Dump the solid waste into a yellow contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the hazardous waste – landfill steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.2. Extracted soil samples, used sodium sulfate, paper funnel filters, glass wool, thimbles, and extracted solids saturated with solvents. Dump these materials into an orange contaminated lab trash bucket. When the bucket is full or after no more than one year, tie the plastic bag liner shut and put the lab trash into the incineration steel collection

drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Waste Methanol. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel flammable solvent drum in the H3 closet. When full to no less than six inches of the top, or after no more than 75 days, move the steel flammable solvent drum to the waste collection area for shipment.

15.4. Mixed water/methanol waste from soil extraction. Collect the waste in the HPLC waste carboy. When full, or after no more than one year, dump into the blue plastic HPLC collection drum in the H3 closet. When the drum is full, to no less than six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.

15.5. Aqueous acidic waste from the LCMS instrument contaminated with methanol. This is collected in a 1-gallon carboy at the instrument. When the carboy is full, or after no more than one year, it is emptied into the blue plastic HPLC collection drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move it to the waste collection area for shipment.

15.6. Autovials contaminated with methanol. As the autovials are removed from the instrument after analysis, they are collected in open containers at the instrument. After all autovials are removed, the open container must be dumped into a closed satellite collection container in a fume hood, as the punctured septa in the autovial can allow methanol and other contaminants to evaporate into the atmosphere. The satellite collection containers are transferred to the waste disposal area when full or after no more than one year, where they are disposed through the vial eater.

16. REFERENCES

16.1. Cheryl Moody, Wai Chi Kwan, Johnathan W. Martin, Derek C. G. Muir, Scott A. Mabury, “Determination of Perfluorinated Surfactants in Surface Water Samples by Two Independent Analytical Techniques: Liquid Chromatography/Tandem Mass Spectrometry and 19FNMR,” Analytical Chemistry 2001, 73, 2200-2206.

16.2. John Giesy et al., “Accumulation of Perfluorooctane Sulfonate in Marine Mammals”, Environmental Science & Technology, 2001 Vol. 35, No. 8, pages 1593-1598.

16.3. U.S. EPA, “Residue Chemistry Test Guidelines, OPPTS 860.1340, Residue Analytical Method”, EPA 712-C-95-174, August 1995.

16.4. STL Denver White Paper DEN-W-LC-002, “Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid

Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, September 5, 2003.

16.5. STL Denver White Paper DEN-W-LC-003, "Addendum A to Method Validation Study for Analysis of Ammonium Perfluorooctanate in Soil Matrices by High Performance Liquid Chromatography/Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, August 6, 2003.

16.6. STL Denver White Paper DEN-W-LC-004, "Method Validation Study for Analysis of Perfluorooctanoic Acid in Waters by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS)", Mark Dymerski, January 26, 2005.

16.7. Waters application note; "Acquity UPLC System for Quantifying Trace Levels of Perfluorinated Compounds with an Acquity PFC Analysis Kit", Peter J. Lee, Evan T. Bernier, Gordon T. Fujimoto, Jeremy Shia, Michael S. Young, and Alice J. Di Gloia, Waters Corporation, Milford, MA. USA.

16.8. US EPA, "Method 537 - Determination of Selected Perfluorinated alkyl acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometery (LC/MS/MS)", Version 1.1, September 2009, J.A. Shoemaker, P.E. Grimmett, B.K. Boutin, EPA Document #: EPA/600/R-08/092

16.9. Erika F. Houtz and David L. Sedlak, "Oxidative Conversion as a Means of Detecting Precursors to Perfluoroalkyl Acids in Urban Runoff," Environmental Science and Technology 46, no. 17 (2012): 9342-49.

17. METHOD MODIFICATIONS

17.1. Modifications from Method 537 are detailed below:

17.1.1. Water sample containers are not preserved with Trizma.

17.1.2. The method has been modified to address soil/solid matrices. The extraction holding time is set at 14 days.

17.1.3. The analyte list has been expanded. The number of labeled analytes has been expanded as well to improve quantitation.

17.1.4. The reporting limits differ as they are all set at one consistent value.

17.1.5. Calibration levels differ from the referenced method.

17.1.6. More labeled analytes are fortified into the samples prior to the extraction process. Most target analytes are quantitated against a labeled analyte.

17.1.7. There is no symmetry requirement.

17.1.8. Calibration, both initial and continuing, has different acceptance criteria due to the longer list of analytes, and the use of isotope dilution quantitation.

17.1.9. The eluents and HPLC configuration differs. As a result the final extract is in 80:20 methanol:water.

17.1.10. The LCS and MS/MSD are spiked at one concentration and do not rotate between a low to high levels.

17.1.11. Samples are not checked for residual chlorine or pH.

17.1.12. A different SPE cartridge (Waters OASIS WAX) is used for the extraction process. As a result solvents and elution procedures are different.

18. ATTACHMENTS

18.1. Attachment 1 - Analysis of Perfluorinated Compounds (PFAS) in Water via In Line Solid Phase Extraction (SPE).

19. REVISION HISTORY

Revisions to Attachment 1 are documented in the attachment.

Revisions prior to 05/01/2017 have been removed and are available in previous versions of this SOP.

19.1. WS-LC-0025, Revision 3.2, Effective 08/20/2018

19.1.1. Section 1 added, “1H,1H,2H,2H-perfluorododecane sulfonate” and “Perfluoro-1-dodecansulfonic acid” entries to table.

19.1.2. Section 1.2 revised table entry for “Adona” to “Dona”.

19.1.3. Section 7.4 added, “PFDoS” and “10:2 FTS” entries to table.

19.1.4. Section 7.4 revised, “Adona” entry to “Dona”.

19.1.5. Table 2 added, “PFDoS”, “PFDoS_2”, and “10:2 FTS” entries to table.

19.1.6. Table 3 revised, “Adona” and “Adona_2” entries to “Dona” and “Dona_2”.

19.1.7. Table 4 added, “PFDoS” and “10:2 FTS” entries to table.

19.1.8. Table 4 revised, “Adona entry to “Dona”.

19.1.9. Editorial changes.

19.2. WS-LC-0025, Revision 3.1, Effective 06/21/2018

19.2.1. Section 11.2.1 revised to, “Visually inspect samples for the presence of settled and/or suspended sediment/particulates. If present or if the sample is biphasic add IDA prior to any sample decanting or centrifugation. If the sample requires decanting or centrifugation contact the client for guidance prior to such action. Decanting or filtering of the sample can lead to a low bias.”

19.2.2. Editorial changes.

19.3. WS-LC-0025, Revision 3.0, Effective 04/13/2018

19.3.1. Section 1.1 updated table with PFPeS and PFNS analytes.

19.3.2. Added Section 2.2, which details the analytes that can be covered by the method under special request.

19.3.3. Added Section 3.13, “AFFF: Aqueous Film Forming Foam”.

19.3.4. Section 6.19 added, “Create all eluents in Reagent module, label eluent containers with TALS label and place 2nd label into maintenance log when put into use” to table.

19.3.5. Section 7.1.2 added, “Prepared by weighing 1.509g of ammonium acetate and dissolving in 1L of water. The resultant solution is filtered through a 0.22um filter before use. This solution has volatile components, thus it should be replaced every 7 days or sooner.”

19.3.6. Section 7.1.3 added, “Prepared by diluting 12mL of ammonium hydroxide into 4L of methanol.”

19.3.7. Section 7.1.8 added, “Prepared by weighing 16g of potassium hydroxide and dissolving in 4L of methanol.”

19.3.8. Section 7.1.11 added, “Prepared by diluting 400mL of 1N NaOH into 3.6L of water for a total volume of 4L.”

19.3.9. Section 7.4 updated table with PFPeS and PFNS analytes.

19.3.10. Section 7.4, added table to detail ICAL for Fluorinated Replacement Compounds.

19.3.11. Added Section 8.1.1, “Water samples collected from a known chlorinated

source should be preserved with Trizma."

19.3.12. Added Section 9.9.3, "If the IS does not meet criteria, re-analyze the extract. If the IS meets criteria in the second analysis, report that analysis. If the IS does not meet criteria in the second analysis, report the first analysis with narration."

19.3.13. Added Section 11.14.6, "Add 2g of potassium persulfate and 1.9 mL of 10N NaOH to each "Post" sample container."

19.3.14. Removed Section 11.14.8, "Add 2g of potassium persulfate and 1.9 mL of 10N NaOH to each "Post" sample container."

19.3.15. Added Section 11.14.9, "Cap each "Post" sample container, invert 2-3 times prior to placing container into water bath."

19.3.16. Added Section 11.5 and associated subsections, which detail the "TOPS (Total Oxidizable Precursor) Assay for Soil Sample".

19.3.17. Section 11.8 updated Table labeling, added PFPeS and PFNS analytes throughout Tables where applicable, and updated Table 7 to reflect current retention times and quantitation.

19.3.18. Section 11.8 added Table 6, "Recommended Instrument Operating Conditions Mass Spectrometer Scan Settings (SCIEX 5500) for Fluorinated Replacement Chemicals"

19.3.19. Section 11.18.3 removed outdated run sequence and replaced with current run sequence.

19.3.20. Editorial changes.

19.4. WS-LC-0025, Revision 2.9, Effective 11/22/2017

19.4.1. Section 1.2, table updated to reflect ranges after removing MeFOSA and EtFOSA from the SOP in the previous revision.

19.4.2. Section 9.3.6, last sentence changed to read, "Reprepare and reanalyze all field and QC samples associated with the contaminated method blank."

19.4.3. Section 9.7, first sentence changed to read, "Initial calibration verification (ICV) – A second source standard is analyzed with the initial calibration curve.

19.4.4. Section 1.3.1 revised to read, "Once the optimal mass assignments (within

±0.5 amu of true) are made immediately following the initial tune, the lowest level standard from the initial calibration curve is assessed to ensure that a signal to noise ratio greater than 10 to 1 (S/N > 10:1) is achieved for each PFAS analyte. The first level standard from the initial calibration curve is used to evaluate the tune stability on an ongoing basis. The instrument mass windows are set initially at ± 0.5 amu of the true value; therefore, continued detection of the analyte transition with S/N > 10:1 serves as verification that the assigned mass remains within ± 0.5 amu of the true value, which meets the DoD/DOE QSM tune criterion. For QSM work, the instrument sensitivity check (section 10.12.4) is also evaluated to ensure that the signal to noise criteria is met."

19.4.5. Editorial changes.

19.5. WS-LC-0025, Revision 2.8, Effective 11/06/2017

19.5.1. Revised Section 4.5 to "Both branched and linear PFAS isomers can potentially be found in the environment. Linear and branched isomers are known to exist for PFOS, PFOA, PFHxS, PFBS, EtFOSAA, and MeFOSAA based upon the literature. If multiple isomers are present for one of these PFAS they might be adjacent peaks that completely resolved or not, but usually with a deflection point resolved during peak integration. The later of these peaks match the retention time of its labeled linear analog. In general, earlier peaks are the branched isomers and are not the result of peak splitting.

At this time only PFOS, PFOA and PFHxS are commercially available as technical mixtures. These reference standards of the technical mixtures for these specific PFAS are used to ensure that all appropriate peaks are included during peak integration."

19.5.2. Sections 4.8 and 7.2.1.1, corrected the in-sample contributions to 0.30 ng/L and 0.015 ug/kg.

19.5.3. Removed Section 7.1.14, "Methanol-Water, 78:22 vol./vol., prepared by mixing 780 mL methanol and 220 mL reagent water. Stored in polypropylene bottle and sealed with polypropylene screw cap." Reagent was added incorrectly.

19.5.4. Section 7.2.4, corrected the factor to 0.956 from 1.046.

19.5.5. Added Section 7.4.1, "A technical (qualitative) grade PFOA standard which contains both linear and branched isomers is used as a retention time (RT) marker. This is used to integrate the total response for both linear and branched isomers of PFOA in environmental samples while relying on the

initial calibration with the linear isomer quantitative standard This technical (qualitative) grade PFOA standard is analyzed initially, after an initial calibration when a new column is installed or when significant changes are made to the HPLC parameters."

19.5.6. Section 9.7, added "Rerun the initial calibration" as the last bullet item.

19.5.7. Added Section 10.3.1, "The first level standard from the initial calibration curve is used to evaluate the tune criteria. The instrument mass windows are set at ±0.5 amu; therefore, detection of the analyte serves as verification that the assigned mass is within ±0.5 amu of the true value, which meets the DoD/DOE QSM tune criterion.

19.5.8. Section 10.10.1, appended "containing both IDA and IS" to the end of the paragraph.

19.5.9. Sections 11.6.3 and 11.12.2.3, changed "78:22 methanol:water" to "methanol".

19.5.10. Sections 1.1 and 7.4, removed EtFOSA and MeFOSA from tables due to low volume of requests for those analytes.

19.5.11. Removed Section 2.2.1, "Optional cleanups may include sample freezing and/or cleanup by SPE cartridge, unless EtFOSA and MeFOSA are requested."

19.5.12. Removed EtFOSA/MeFOSA specific comments in various sections throughout the document.

19.5.13. Section 7.4 Note added, "The concentration of the calibration solutions for non-concentrated extracts is 1/20th the levels indicated above."

19.5.14. Section 7.9, changed 1000 ng/mL to 250 ng/mL and replaced final sentence with "The internal standard solution used for the non-concentrated extracts is at a concentration of 50 ng/mL."

19.5.15. Removed Section 11.2.8, "If EtFOSA and/or MeFOSA are requested, add 100uL of IS and then adjust the final volume (FV) of these aliquots to 5.0 mL with MeOH. QC samples, LCS, MS, and MSD will require concentration via nitrogen to adjust the FV to 5.0 mL. Vortex each sample. Then, transfer a portion of the extract to a 300 uL polypropylene autosampler vial (7 drop-wise or approximately ½ filled is sufficient). Archive the rest of the extracts for re-injection and dilution."

19.5.16. Added Section 11.5.4, "Proceed to Section 11.15.2 (Graphitized Carbon

Cleanup) as needed. This is required for all DoD/DOE extracts."

19.5.17. Added Section 11.7.1.1, "Seal the test tube tightly. Invert container several times and then vortex. Allow extract to settle for 10 minutes prior to moving to the next step."

19.5.18. Inserted Section 11.8.1.1, "Projects performed under the auspices of the DoD/DOE must have the entire sample homogenized prior to subsampling in accordance with QSM 5.1 criteria."

19.5.19. Section 11.11.4, added "(Graphitized Carbon Cleanup) as needed. This is required for all DoD/DOE extracts."

19.5.20. Section 11.14.6, added "Spike all "Pre" and "Post" samples with 25uL of the reverse surrogate solution (Section 7.8)."

19.5.21. Section 11.15.2, revised to read, "Cleanup with graphitized carbon will be applied to all samples as needed but is required for all DoD/DOE extracts."

19.5.22. Added Section 11.15.2.5, "Proceed to Section 11.6, 11.7, or 11.12 as applicable."

19.5.23. Removed Sections 11.15.3 through 11.15.6.

19.5.24. Added Section 11.16, "AFFF Sample Preparation".

19.5.25. Section 11.17, removed EtFOSA, MeFOSA, d5-EtFOSA, and d3MeFOSA from all tables.

19.5.26. Section 11.17, changed masses for M2-4:2FTS, M2-6:2FTS, and M2-8:2FTS. Initially assigned daughter masses were bleeding through from the native analog.

19.5.27. Section 11.17, all tables on MS Interface Mode Line, added "Minimum of 10 scans/peak."

19.5.28. Added Section 11.17.1, "Post Spike Sample Analysis for AFFF Samples".

19.5.29. Added Section 11.8.4.1 "Spike non-concentrated samples at 0.5 mL of LCS/Matrix Spike Solution."

19.5.30. Added Section 11.8.5.1, "Spike non-concentrated samples at 0.5 mL of IDA PFC Solution."

19.5.31. Editorial changes.

19.6. WS-LC-0025, Revision 2.7, Effective 09/20/2017

19.6.1. Section 1.1 table, added 1H,1H,2H,2H-perfluorohexane sulfonate *(4:2)*.

19.6.2. Section 1.1, removed "Sample results for PFOA may also be reported as APFO, at the request of the client. (See Section 12.7)."

19.6.3. Section 1.2 and 11.8.2, updated tissue extracted mass and RL.

19.6.4. Section 2.5, removed "and assumes a proportional relationship between the initial calibration and the analyte in the extract. The ratio of the peak response to mass or concentration injected is used to prepare a calibration curve."

19.6.5. Added Section 6.6, "Extract concentrator or nitrogen manifold with water bath heating to 50-55°C".

19.6.6. Added Section 7.1.14, "Methanol-Water, 78:22 vol./vol., prepared by mixing 780 mL methanol and 220 mL reagent water. Stored in polypropylene bottle and sealed with polypropylene screw cap."

19.6.7. Section 7.2.1.1, revised "roughly 0.15 pg/L" to "roughly 0.15 ng/L".

19.6.8. Section 7.4 table, added:

4:2 FTS	0.5	1.0	2.0	20	50	200	400

19.6.9. Section 7.4 table, revised Labeled Isotope Dilution Analytes (IDA) Section.

19.6.10. Section 7.4 table, added:

Internal Standard (IS)							
13C2-PFOA	50	50	50	50	50	50	50

19.6.11. Section 7.4, removed "FOSAA may be added to the mix and are added at the same concentration as FOSA."

19.6.12. Added Section 7.9, "Internal Standard Solution, 1000 ng/mL. The internal standard solution is prepared by diluting 13C2-PFOA to produce a solution containing this compound at a concentration of 1000 ng/mL in methanol. This is added to all extracts prior to analysis. Non-concentrated extracts are fortified with a 5X dilution of this solution."

19.6.13. Section 8.1, changed "250 mL" to "8 oz."

19.6.14. Added Sections 9.3.6, 9.8.2.3, 10.10.4, 10.8.2.5, 10.11.3, and 10.12.4 to address DOD QSM 5.1 Table B-15 criteria.

19.6.15. Added Section 9.9, “Internal Standard.”

19.6.16. Updated all tables to indicate target analyte quantitation via isotope dilution. Internal standard quantitation is only used to quantitate the IDA recoveries.

19.6.17. Added Section 10.8.2.4, 10.12.2, and 10.12.2.1 to incorporate IS criteria into calibrations.

19.6.18. Section 11.2.1, “Evaluate if the sample can be decanted or centrifuged; if not, contact the client for guidance. Filtering the sample can lead to a low bias.”

19.6.19. Added Section 11.2.3.1, “Alternatively, weigh the sample container prior to extraction and then weigh the sample container after extraction to determine the initial volume.”

19.6.20. Added Section 11.5.3, “Note: If the extracts will not be concentrated elute extract with a total of 8 mL of 0.3% NH_4OH/methanol.”

19.6.21. Added Section 11.6.2.3, “Add 300 uL of the 78:22 methanol:water solution and mix the contents well using a vortex mixer.”

19.6.22. Added Section 11.6.2.4, “Add 100 uL of Internal Standard (IS) solution to each extract and vortex to mix.”

19.6.23. Added Section 11.7, “Final volume for non-concentrated extract”.

19.6.24. Revised Section 11.11, “SPE Elution of Solid Extracts”.

19.6.25. Revised Section 11.12, “Extract Concentration for Solid Samples”.

19.6.26. Removed Section 12.8, “If results are to be reported as ammonium perfluorooctanoate (APFO), instead of PFOA, apply a multiplier of 1.0406 to the sample results to correct for the molecular weight differences between PFOA and APFO or this adjustment can be made during the preparation of the standards used for calibration. (Use one, not both.)”

19.6.27. Removed Section 13.4 – it was a copy of Section 13.2.

19.6.28. Various revisions to fulfill requirements based on DOD/DOE QSM 5.1.

19.6.29. Editorial changes.

19.7. WS-LC-0025, Revision 2.6, Effective 08/15/2017

19.7.1. Section 7.4, added MPFBS, MPFTeDA, and MPFHxDA to the table.

19.7.2. Section 11.15, added 13C-PFBS to the Recommended Instrument Operating Conditions table for SCIEX 5500.

19.7.3. Section 11.15 Recommended Instrument Operating Conditions table, changed the mass transitions for native PFTeDA from 713 > 669 (quant) and 713 > 169 (qualifier) to 713 > 169 (quant) and 713 > 219 (qualifier).

19.7.4. Editorial changes.

19.8. WS-LC-0025, Revision 2.5, Effective 07/10/2017

19.8.1. Revised Section 11.6.1 to read “Prior to concentrating each sample, add 100 uL of water.”

19.8.2. Revised Section 11.6.2 to read “Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 100 uL of water remains.

11.6.2.1 This blow down must take a minimum of 3.5 hours.

11.6.2.2 Extracts can not remain in the water bath longer than 5 minutes once concentrated.”

19.8.3. Revised Section 11.6.3 to read “Add 400 uL of methanol to each extract, soak, and vortex to mix well. This will create an extract with a final solvent composition of 80:20 methanol:water.”

19.8.4. Revised Section 11.11.1 to read “Prior to concentrating each sample, add 200 uL of water.”

19.8.5. Revised Section 11.11.2 to read “Concentrate each sample under a gentle stream of nitrogen until the methanol is evaporated and the 200 uL of water remains.”

11.11.2.1 This blow down must take a minimum of 3.5 hours.

11.11.2.2 Extracts can not remain in the water bath longer than 5 minutes once concentrated.”

19.8.6. Revised Section 11.11.3 to read “Add 800 uL of methanol to each extract, soak, and vortex to mix well. This will create an extract with a final solvent composition of 80:20 methanol:water.”

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of water samples via in line solid phase extraction (SPE) for the following compounds using liquid chromatography / tandem mass spectrometry (LC/MS/MS) on a SCIEX 5500.

Compound Name	Abbreviation	CAS #
Perfluoroalkylcarboxylic acids (PFCAs)		
Perfluoro-n-heptanoic acid	PFHpA	375-85-9
Perfluoro-n-octanoic acid	PFOA	335-67-1
Perfluoro-n-nonanoic acid	PFNA	375-95-1
Perfluorinated sulfonic acids (PFSAs)		
Perfluoro-1-butanesulfonic acid	PFBS	375-73-5
Perfluoro-1-hexanesulfonic acid	PFHxS	355-46-4
Perfluoro-1-octanesulfonic acid	PFOS	1763-23-1

1.2. The working range of the method is listed below. The linear range can be extended by diluting the extracts.

Matrix	Nominal Sample Size	Reporting Limit	Working Range
Water	1.0 mL	2.0 ng/L	2 to 200 ng/L

2. SUMMARY OF METHOD

2.1. A 1 mL aliquot of sample is diluted to a 40:60 methanol:water extract and analyzed by LC/MS/MS. PFAS are separated from other components on a C18 column with a solvent gradient program using 20mM ammonium acetate/water and methanol.

3. DEFINITIONS

Refer to Section 3 of the main body of this SOP for a summary of definitions.

4. INTERFERENCES

Refer to Section 4 of the main body of this SOP for interferences.

5. SAFETY

Refer to Section 5 of the main body of this SOP for safety information.

6. EQUIPMENT AND SUPPLIES

Refer to Section 6 of the main body of this SOP for supplies, other than those listed below specific to the in line SPE analysis.

6.1. 2 mL auto sampler vials, clear glass, Thermo Scientific Nation surestop vial, part no. C5000-1, or equivalent**.**

6.2. Vial caps, Thermo Scientific National AVCS blue cap, pre slit TEF/STL septa, part no. C5000-55B or equivalent.

6.3. Eppendorf 1000 uL epTIPS, part no. 022491954 or equivalent.

6.4. Eppendorf 200 uL epTIPS, part no. 022491938 or equivalent.

6.5. 50 mL graduated plastic centrifuge tubes, SCP Science DigiTUBES part no. 010-500-263 or equivalent

6.6. 1000 uL Pipette: Eppendorf Research Plus

6.7. 100 uL Pipette: Rainin EDP3-Plus

6.8. 250 mL HDPE bottles with PPE screw caps, ESS part no. 0250-1902-QC or equivalent.

6.9. Analytical columns

6.9.1. Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm, Part No. 00D-4439-Y0, or equivalent.

6.9.2. PFAS Isolator column, Phenomenex Luna C18 5 um, 50 mm x 4.6 mm, part no. 00B-4252-E 0 or equivalent.

6.10. SCIEX 5500 Triple Quad MS. The system utilizes Chrom Peak Review, version 2.1 or equivalent.

6.11. Shimadzu CTO-20AC HPLC equipped with 3 LC-20AD pumps and one DGU-20 degassing unit or equivalent.

7. REAGENTS AND STANDARDS

Refer to Section 7 of the main body of this SOP for reagents and standards, other than those listed below specific to the in line SPE analysis.

7.1. Reagent grade chemicals shall be used in all tests whenever available. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on the Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1. Ammonium acetate, Fisher Optima LCMS grade (20 mM in water), part no. A114-50, or equivalent.

7.1.2. Methanol, Baker HPLC grade, part no. 9093-03.

7.1.3. Water, Nanopure or Millipore or Fisher Optima LCMS grade, part no. W6-4, must be free of interference and target analytes.

7.2. Calibration Standards

The calibration stock solution is prepared by diluting the appropriate amounts of the stock solutions (Section 7.2 of the main body of this SOP) in 40:60 methanol:water. The calibration stock solution is diluted with methanol to produce initial calibration standards. These are the normal calibration levels used. A different range can be used if needed to achieve lower reporting limits or a higher linear range.

7.3. Initial Calibration (ICAL) Levels (ng/L)

Compound	CS-1	CS-2	CS-3	CS-4	CS-5	CS-6	CS-7	CS-8
Perfluoroalkylcarboxylic acids (PFCAs)								
PFHpA	1.0	2.0	5.0	10	20	50	100	200
PFOA	1.0	2.0	5.0	10	20	50	100	200
PFNA	1.0	2.0	5.0	10	20	50	100	200
Perfluorinated sulfonic acids (PFSAs)								
PFBS	1.0	2.0	5.0	10	20	50	100	200
PFHxS	1.0	2.0	5.0	10	20	50	100	200
PFOS	1.0	2.0	5.0	10	20	50	100	200
Labeled Isotope Dilution Analytes (IDA)								
13C4-PFHpA	50	50	50	50	50	50	50	50
13C4-PFOA	50	50	50	50	50	50	50	50
13C5-PFNA	50	50	50	50	50	50	50	50
18O2-PFHxS	50	50	50	50	50	50	50	50
13C4-PFOS	50	50	50	50	50	50	50	50

Note- The above calibration levels are provided only as an example. The actual ICAL level used for each analytical batch will depend upon the LOQ requirements of the program.

7.4. LCS/Matrix PFC Spike Solution, 100 ng/mL.

The PFC spike solution is prepared by diluting all PFAS to produce a solution containing each PFAS at 100 ng/mL in methanol.

7.5. PFC Isotope Dilution Analyte (IDA) Spike Solution, 1 ng/mL.

The PFC-IDA solution is prepared by diluting all labeled PFAS to produce a solution containing each at 1 ng/mL in methanol.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Water samples are collected in pre-cleaned 250 mL HDPE containers. Other containers may also be suitable. Samples are chilled to 0 - 6 °C for shipment to the laboratory.

8.2. Samples are logged in following normal laboratory procedures and are stored under refrigeration at 0 - 6 °C. Water samples must be analyzed within 28 days of collection.

9. QUALITY CONTROL

Refer to Section 9 of the main body of this SOP for Quality Control information.

9.1. If potable water samples from the state of New York (NY) are analyzed via this method the control limits for LCS and IDA for PFOS and PFOA recoveries are 70-130%. If these limits are not met, refer to Section 9 of the main body of this SOP for corrective action.

9.2. If POST (treatment) samples have positive detections, review the associated PRE and MID (treatment) samples for similar detections. Re-preparation and re-analysis may be needed.

9.3. If PFBS is detected in the method blank greater than the RL, evaluate data for impact. PFBS is a known laboratory artifact. Re-preparation and re-analysis may be needed.

10. CALIBRATION

Refer to Section 10 of the main body of the SOP for calibration information.

11. PROCEDURE

Refer to Section 11 of the main body of this SOP for procedures, other than those listed below specific to the in line SPE analysis.

11.1. Water Sample Preparation

11.1.1. Visually inspect samples for the presence of settled and or suspended sediment/particulate. Evaluate if the sample can be decanted or centrifuged; if not, contact the client for guidance. Filtering the sample can lead to a low bias.

If authorized by the client to filter the sample, filter the water sample through a glass fiber filter (Whatman GF/F Cat No 1825 090 or equivalent). Gravity of vacuum can be used to pass the sample through the filter. Prepare a filtration blank with any samples requiring filtration. File an NCM noting the need for filtration.

Warning: The use of a vacuum system creates the risk of glassware implosion. Inspect all glassware prior to use. Glassware with chips, scratches, rub marks or cracks must not be used.

11.1.2. Prepare an LCS and method blank by adding 250 mL of HPLC grade water into a 250 mL HDPE bottle.

11.1.3. If requested, find the client assigned sample for MS/MSD.

11.1.4. Spike directly into the sample bottles for the LCS and MS/MSD (if requested) with 0.050 mL (50 uL) of the LCS/Matrix PFC Spike solution (Section 7.4). This will result in a sample concentration of 20 ng/L. Shake well to disperse spike.

11.1.5. Measure 1 mL of each sample using an Eppendorf pipette and pour into a labeled 2.0 mL injection vial. This includes the LCS and method blank samples as well.

11.1.6. Be sure to "prepare" the pipette by collecting two 1 mL aliquots and disposing of them, and then collect the aliquot for testing.

11.1.7. Add 83 uL of surrogate solution (PFC IDA Spike Solution, Section 7.5) into each vial for each sample and QC sample. This will result in an extract concentration of 50 ng/L for the surrogate.

11.1.8. Add 577 uL of methanol to each sample for a final solvent composition of 40:60 methanol:water.

11.1.9. Seal the vial with a polypropylene screw cap. Note: Teflon lined caps can not be used due to detection of low level concentration of PFAS.

11.1.10. Vortex to mix the mixture well.

11.2. Instrument Analysis

11.2.1. Suggested operation conditions are listed in Tables 1A-1C below:

Table 1A - Routine Instrument Operating Conditions					
HPLC Conditions (Shimadzu HPLC)					
Column (Column temp = 35°C)	Phenomenex Gemini C18 3 um, 3.0 mm x 100 mm				
Mobile Phase Composition	A = 20 mM Ammonium Acetate in Water B = Methanol				
Gradient Program	**Time (min)**	**%A**	**%B**	**Curve**	**Flow Rate (mL/min)**
	0	90	10	6	0.60
	1	90	10	6	0.60

Table 1A - Routine Instrument Operating Conditions					
HPLC Conditions (Shimadzu HPLC)					
	1.5	35	65	6	0.60
	8	5	95	6	0.60
	8.1	1	99	6	0.60
	12	1	99	6	0.60
	12.5	90	10	6	0.60
	Maximum Pressure limit = 5,000 psi				
Injection Size	950 uL (fixed amount throughout the sequence)				
Run Time	17.1 minutes				
MS Interface Mode	ESI Negative Ion. Minimum of 10 scans/peak.				
Ion Spray Voltage (kV)	4.5				
Entrance Potential (V)	5				
Declustering Potential (V)	25				
Desolvation Temp	550 ºC				
Curtain Gas (nitrogen) Flow	35 psi				
Collision Gas (nitrogen) Flow	8 psi				

Table 1B - Routine Instrument Operating Conditions						
Mass Spectrometer Scan Settings (SCIEX 5500)						
Compound	**Comments**	**Reaction (MRM)**	**Dwell (sec)**	**Ent. Pot. (V)**	**Col. Energy (V)**	**Declu. Pot. (V)**
PFBS	Perfluorobutanesulfonate	299 > 80	0.02	6	58	55
18O2-PFHxS	IDA	403 > 84	0.02	12	74	60
PFHpA	Perfluoroheptanoic acid	363 > 319	0.02	6	12	25
13C4-PFHpA	IDA	367 > 322	0.02	6	12	25
PFHxS	Perfluorohexanesulfonate	399 > 80	0.02	12	74	60
18O2-PFHxS	IDA	403 > 84	0.02	12	74	60
PFOA	Perfluorooctanoic acid	413 > 369	0.02	6	14	25
13C4PFOA	IDA	417 > 372	0.02	6	14	25
PFNA	Perfluorononanoic acid	463 > 419	0.02	6	14	25
13C5-PFNA	IDA	468 > 423	0.02	6	14	25
PFOS	Perfluorooctanesulfonate	499 > 80	0.02	9	108	65
13C4-PFOS	IDA	503 > 80	0.02	9	108	65

Table 1C				
Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFBS	6.68	18O2-PFHxS	7.76	Isotope Dilution
PFHpA	7.77	13C4-PFHpA	7.77	Isotope Dilution

Table 1C				
Native Compounds	**Typical Native RT (minutes)**	**IS analog**	**Typical IDA RT (minutes)**	**Quantitation Method**
PFHxS	7.76	18O2-PFHxS	7.76	Isotope Dilution
PFOA	8.44	13C4-PFOA	8.44	Isotope Dilution
PFNA	9.10	13C5-PFNA	9.10	Isotope Dilution
PFOS	9.06	13C4-PFOS	9.06	Isotope Dilution

11.2.2. Tune and calibrate the instrument as described in Section 10.

11.2.3. A typical run sequence is as follows:

- Primer (A number of primers are injected for conditioning of the instrument before analysis, especially when the instrument was idled or changed from a different analysis).
- Blank
- Calibration Curve
- ICB
- ICV
- PFOA RT marker (as needed)
- Rinse Blank (RB, not linked to anything)
- MB
- LCS
- LCSD (if applicable)
- Sample 1
- Sample 1 MS (if applicable)
- Sample 1 MSD (if applicable)
- Sample 2 (up to sample 10 before next CCV)
- CCV
- Up to 10 samples.
- End sequence with CCV

12. CALCULATIONS

Refer to Section 12 of the main body of this SOP for calculation information.

13. METHOD PERFORMANCE

Refer to Section 13 of the main body of this SOP for method performance information.

14. POLLUTION PREVENTION

Refer to Section 14 of the main body of this SOP for pollution prevention information.

15. WASTE MANAGEMENT

Refer to Section 15 of the main body of this SOP for waste management information.

16. REFERENCES

Refer to Section 16 of the main body of this SOP for reference information.

17. METHOD MODIFICATIONS

17.1. Refer to Section 17 of the main body of this SOP for modifications from Method 537, except as detailed below:

17.1.1. Water samples are prepared at 1.0 mL, not 250 mL.

17.1.2. Water sample containers are not preserved with Trizma. Holding time has been changed to 28 days for analysis.

17.1.3. The eluents and HPLC configuration differs. As a result the final extract is in 40:60 methanol:water.

18. ATTACHMENTS

There are no attachments to this Appendix.

19. REVISION HISTORY

Revisions prior to 04/10/2017 have been removed and are available in previous versions of this SOP.

19.1. WS-LC-0025, Attachment 1, Revision 3.0, Effective 04/13/2018

19.1.1. Updated labeling and formatting of Tables 1A-1C.

19.1.2. Added section 11.2.3, detailing a typical run sequence.

19.2. WS-LC-0025, Attachment 1, Revision 2.9, Effective 11/27/2017

19.2.1. No changes to the attachment with this revision.

19.3. WS-LC-0025, Attachment 1, Revision 2.8, Effective 11/06/2017

19.3.1. Section 11.2.1, Routine Instrument Operating Conditions table (SCIEX 5500), added “Minimum of 10 scans/peak”.

19.4. WS-LC-0025, Attachment 1, Revision 2.7, Effective 09/22/2017

19.4.1. Section 6.5, removed "The 5 items above are to be maintained in the drawer labeled "Segregated Supplies for in line SPE Analysis" in the LC/MS instrument room."

19.4.2. Added Sections 9.1 – 9.3.

19.4.3. Updated Section 11.1.

19.4.4. Editorial changes.

19.5. WS-LC-0025 Attachment 1, Revision 2.6, Effective 08/11/2017

19.5.1. No revisions to this attachment.

19.6. WS-LC-0025 Attachment 1, Revision 2.5, Effective 07/10/2017

19.6.1. No revisions to this attachment.

19.7. WS-LC-0025 Attachment 1, Revision 2.4, Effective 04/25/2017

19.7.1. No revisions to this attachment.

19.8. WS-LC-0025 Attachment 1, Revision 2.3, Effective 04/10/2017

19.8.1. Changed all mentions of "direct aqueous injection (DAI)" to "in line solid phase extraction (SPE)."

19.8.2. Inserted Section 17.1, and changed formatting of the modifications to Method 537 to Section 17.2 and subheadings.

Appendix C

Historical Reports

The following historical reports are provided on the electronic (CD) version of this report.

Final UFP-QAPP

Draft 1,4-dioxane Groundwater Investigation Work Plan

Draft Work Plan for the PFAS Expanded Site Investigation, SEAD 25 and SEAD 26, Seneca Army Depot Activity

Appendix D

Resumes

Years of Experience:

17

Location:

Boston, Massachusetts

Education:

- Bachelor of Science, Chemical Engineering, Brown University

Registrations/Licenses:

- Engineer in Training

Special Training:

- Project Management Certification, Parsons
- 40-hour Hazardous Waste Operations Course, OSHA
- 8-hour Supervisory Instruction Course, OSHA
- Parsons PACE Quality Management Program
- Radiological Fundamentals Training Course (Parsons)
- Certified Grade 2 Industrial Wastewater Treatment Plant Operator

Beth Badik

Senior Project Manager

Summary of Relevant Qualifications

Ms. Beth Badik has 17 years of experience in managing Military Munitions Response Program (MMRP) and hazardous waste site projects on federal and industrial sites, involving remedial investigation (RI), Feasibility Study (FS), remedial design, remedial action construction oversight, groundwater and treatment system startup and operation. She is experienced in managing performance based task orders (TOs), and managing multiple sites within a single project.

Relevant Project Experience

Program Manager / Project Manager for Seneca Army Depot (Seneca), Romulus, New York. Manages over $20M for nine task orders for more than 12 sites at Seneca for the Army Corps of Engineers. Projects have been both fixed-price and T&M contracts. Scope elements include munitions response removal actions, digital geophysical investigations, soil sampling, long-term groundwater monitoring, excavation of impacted soil or source areas totally 60,000 cy of soil, implementation and monitoring of a bioremediation groundwater remedy, and preparation of CERCLA documents (RIs, FSs, Proposed Plans, RODs/Decision Documents, Work Plans, and Closure Reports).

An example project includes the Open Detonation (OD) Grounds RI/FS where Ms. Badik is managing a $5M project to characterize and address impacts from munitions and explosives of concern (MEC) and metals and explosives in soil. Managed the preparation of the nature and extent memorandum characterizing the chemical impacts and the potential munitions impacts on the site. Oversaw the human health risk assessment and m hazard analysis. Prepared the FS, evaluating options to address the munitions hazards and the impacts of metals to soils including excavation, digital geophysical mapping (DGM), “mag and dig”, capping, and sifting. Prepared work plan for remedial action covering reacquisition and mag and dig munitions response activities. Munitions field effort to reacquire and investigate 14,700 anomalies and mag and dig 60 acres, removing 244 MEC items and 106 tons of certified scrap metal. Provided munitions safety training and a UXO technician escort for the earth work contractor to support their work. Completed work over three field seasons with a staff of up to 23 people onsite at one time, with no health and safety incidents.

Another example is the Ash Landfill Bioremediation project, where Ms. Badik is managing an ongoing groundwater LTM program at the Ash Landfill site for VOCs (specifically trichloroethene) under three consecutive FFP task orders. The LTM goal is to demonstrate the effectiveness of a series of mulch biowalls on the biodegradation of the TCE plume. LTM program includes 14 wells sampled semiannually. Prepared a letter demonstrating to EPA that the remedy has achieved Operating Effectively and Successfully (OPS) status. Prepared

semiannual letter reports, annual reports, and remedy evaluations. Responsible for staffing, managing the budget and schedule, and coordination with the client. FFP project continues to exceed its GP goal. Currently preparing work plan to implement recharge of the biowall system to maintain the OPS determination.

Project Manager for MMRP Source Removal Action at Joint Base Cape Cod, Massachusetts. Ms. Badik manages this project with CEHNC, the National Guard Bureau, and CENAE at JBCC. Ms. Badik oversaw the preparation of the PMP, UFP-QAPP, and the required DAGCAP plans for advanced geophysical classification. She is planning field work to conduct dynamic AGC surveys over 10 acres, cue anomalies, and investigate the selected anomalies to achieve the removal goals.

Program Manager for Baltimore MAMMS II Contract. Manages the relationship between Parsons and USACE Baltimore District. Coordinates responses to task order RFPs and sources sought. Initiates and attends client meetings and brown bag presentations provided to the district by Parsons.

Project Manager for Lake City Army Ammunition Plant – Demolition of Explosively-Contaminated Facilities (MEGA TO) and EE/CA for Building 83 (WERS TO), Independence, Missouri. For the MEGA TO, Ms. Badik oversaw the development of the ESS, Work Plan, and the UFP-QAPP for the demolition and remediation of 31 explosively contaminated facilities. The field work is planned for Spring 2018.

For the WERS TO, Ms. Badik developed engineering evaluation/cost analysis (EE/CA) and a work plan to address a building coated in explosives residue with asbestos containing materials. EE/CA identified possible proactive actions to address safety concerns to minimize likelihood of a high order explosion from the explosive risk. Evaluated innovative technologies, including thermal convection systems and chemical deactivation products. Prepared Work Plan that proposes use of new chemical activation product, MuniRem, to neutralize the explosives, allow for standard asbestos abatement, followed by building demolition. Planned the metals and explosives confirmation soil sampling following building demolition. Supports community relations by serving as lead contact for Army client with state regulator and EPA to address concerns and explain the Army's technical approach. Attended a RAB meeting and supporting the installation and Army Corps by preparing presentation materials.

Project Manager, **Munitions Response Actions at Former Brunswick Naval Air Station in Brunswick, Maine.** Managing two task orders covering three sites (former OB/OD Grounds, Quarry, and skeet ranges) to address munitions and metals in soil. Developed work plans for investigations at multiple skeet ranges. Supports Navy with communication with the regulators. Prepared presentations for Army/Regulatory meetings and RABs to explain the technical approach

and receive buy-in.

Designed multi-phased pre-design sampling program to reduce costs and expedite field work. Oversaw investigations, and evaluated data to delineate lead-impacted excavation area for the design. Oversaw the remedial action and lead real-time decision making on the extent of excavation required to meet clean up goals. Developed plan for a wetlands delineation and a biological survey at a pond at a former OB/OD site.

Conducted DGM surveys at Site 12 in support of two remedial actions. Developed innovative field method to complete DGM work in a drained pond where typical survey methods were not suitable due to uneven and unstable terrain that prohibited traversing the pond on foot. Approach resulted in achieving full coverage with no health or safety incidents, schedule delays, or budget impacts.

Preparing a FS that covers potential impact from radiological, munitions, and Hazardous and Toxic Waste (HTW) concerns. Alternatives under evaluation include “mag and dig”, capping, sifting, and source removal for a TCE plume.

Project Manager for NAVFAC Washington TOs in Maryland and Virginia. Project Manager for three separate TOs for the Navy to complete Munitions Support work at Naval Air Station Patuxent River (Pax River) in Maryland, and Marine Corps Base Quantico, Virginia. Coordinating construction support activities with Pax River by providing UXO technicians (UXO Tech IIs) to serve as escorts for a third-party contractor’s work conducting geotechnical soil borings and construction activities in areas with potential munitions impacts. Responsible for the safety of personnel and ensuring no exposure to munitions.

Manage TOs at Quantico for operational range clearance activities. Providing UXO technicians to conduct a surface clearance, vegetation removal (through a combination of brush cutting, herbicide application, and aerial burning), and target removal/ replacement. Work included radiological surveys of targets. Coordinate staffing to provide UXO escorts for safety during site visits on the ranges.

BETH DRISKILL
SENIOR SCIENTIST

Experience Summary

Analytical and environmental testing, data validation, quality assurance and quality control, writing Quality Assurance Project Plans (QAPP), including Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP), Sampling Plans, and Work Plans for various projects, project/sampling coordination, laboratory procurement, analytical laboratory audit, resolving laboratory non-conformance issues, data interpretation, and data validation.

Years of Experience:

Over 15 years of analytical laboratory and chemistry-related experience.

Years with Parsons:

4 years, 6 months

Education

Certificate of Biotechnology, May 2001, Austin Community College in Austin, Texas

Professional Training:

Advanced Quality Systems for Chemical Analysis, The NELAC Institute, 2007.

Primary Experience

June 2013 – Present Parsons, Senior Scientist. Responsible for laboratory procurement, project quality control and project field sampling coordination between Parsons and subcontracting laboratories, overseeing laboratories performance including the handling of laboratory non-conformance issues, tracking of sample shipments from the field to the laboratory, verifying laboratory receipt and log-in information, tracking data package due dates and delivery dates, organizing incoming data packages, performing laboratory data interpretations and verification/validation of laboratory reports, organizing and archiving completed data sets, reviewing laboratory documentation for compliance with project requirements, electronic data deliverable validation through ADR, preparing Quality Assurance Project Plans (QAPP), including Uniform Federal Policy for Quality Assurance Project Plans (UFP_QAPP), Sampling Plans, and Work Plans for various projects.

Other Experience

August 2001 – May 2003 Severn Trent Laboratories (STL), Analyst I. Organic Preparation Laboratory- Responsible for preparation of sample extracts for organic analyses. Duties included extraction, concentration, and cleanup of aqueous and solid samples based on SW-846 and EPA methodologies and QA/QC protocols. Prepared standards and general laboratory paperwork such as tracking samples, bench sheets, and laboratory logbooks.

May 2003 – October 2003 Severn Trent Laboratories (STL), Supervisor I. Organic Preparation Laboratory- Provided technical and operational support to the laboratory area and oversaw the responsibilities of analysts and

technicians to ensure appropriate testing procedures were in compliance with QA and SOP requirements. Duties included scheduling and prioritizing work tasks, training employees and managed all in house projects including coordinating work projects with project managers.

October 2003 – November 2004 Severn Trent Laboratories (STL), Analyst II. Responsible for the preparation and analysis of environmental samples using routine and complex testing by GC determination of BTEX and gasoline by EPA method 602, 8021B, 8015B GRO, Iowa OA-1 and Tennessee GRO. Other duties included uploading data into reporting system, reviewed data, prepared standards, and the operation, calibration and maintenance of laboratory instruments.

November 2004 –September 2010 TestAmerica Laboratories (formerly Severn Trent Laboratories (STL)) Quality Assurance Specialist. Assisted the QA Manager with internal and external audits and client visits, identified systematic problems within the laboratory and maintaining/updating laboratory reference data in the LIMS. Other duties included assisting operations in writing laboratory SOPs, reviewed non-conformances, performing QA data audits, reviewed/validated laboratory data, sample log-in, managed and updated proposal information, and data package preparation.

September 2010 –January 2011 TestAmerica Laboratories, Project Management Assistant. Coordinated and monitored project scheduling, timely completion and maintenance of project documentation files and completion of project set up. Other essential duties included collating data reports and CLP-like data packages for delivery to clients and reviewed for accuracy, writing case narratives accompanying data packages to communicate anomalies to the client, monitored report due dates for timely delivery, sample login and review of sample login.

January 2011 – June 2013 TestAmerica Laboratories, Semivolatiles Supervisor. Provided technical and operational support to the semivolatiles laboratory and coordinating work projects with project managers to appropriately prioritize laboratory workload to meet client needs. Other responsibilities include scheduling and prioritizing work tasks, training, problem solving, implementing new procedures and methods, analyzing samples, uploading data to reporting system and reviewing and validating data.

PARSONS

envision more

Todd Belanger

SENIOR GEOLOGIST

SUMMARY OF RELEVANT QUALIFICATIONS

Mr. Todd Belanger has experience in a broad range of HTW and MMRP environmental projects including long-term monitoring of sites with groundwater contamination (e.g., PFAS, TCE, BTEX, explosives, metals, perchlorate), military munitions response program investigations (site investigations, munitions removals, RI/FS to ROD process), remedial system installation and monitoring (bioreactor, permeable reactive barriers), and project and field management. His experience includes report writing, field team management, environmental field experience, and project management tasks. In the future, Todd is interested in the implementation of improved visualization techniques to provide clients/regulators with more innovate deliverables, the increased use of research methods and innovative approaches to develop remedial solutions, and investigating the potential use for real-time data collection in the field (e.g., use of tablets and GIS in the field). Todd keeps up to date on industry research by attending webinars (e.g., ESTCP, EPA CLU-IN), Parsons environmental subject matter groups, and working with company subject matter experts.

RELEVANT WORK EXPERIENCE

Deputy Project Manager/Geologist/Field Manager. Long-Term Monitoring Sites, Seneca Army Depot (SEDA), Romulus, NY. Todd is responsible for the day-to-day management of these projects which includes a variety of roles related to several groundwater remediation projects at the Depot. Todd has provided contracting and project management support, office-based field team support, and report preparation for eight sites with groundwater contamination. Investigated COCs include metals, BTEX, per- and polyfluoroalkyl substances (PFAS), and chlorinated VOCs. The investigation at the PFAS sites included direct push drilling and one of few groundwater sampling studies of these compounds within the company. Todd is a company-wide leader in understanding PFAS groundwater sampling and drilling methodologies, since this work required an innovative approach due to highly restrictive sampling requirements (e.g., restrictions on the type of sampling equipment, sampler clothing and hygiene requirements). The chlorinated VOC site includes the performance monitoring of permeable mulch biowalls, the monitoring of reductive dechlorination and the refresh of organic carbon content within the mulch biowalls with an injected substrate (emulsified vegetable oil [EVO] and pH buffer). [2013 – Current]

Deputy Project Manager/Geologist. Various Munitions Sites Seneca Army Depot (SEDA), Romulus, NY. This project involves the closure and land transfer of several former munitions sites within SEDA. Todd is responsible for the production of various documents associated with the site investigation, remedial investigation/feasibility study, proposed plan, and record of decision for these sites. Unique requirements for the closure of an open detonation area have led to a close relationship with the client and federal and state regulators. [2013 – Current]

Deputy Project Manager/Geologist. ConEdison Former MGP Site, Yonkers, NY. Todd is responsible for the management of a NAPL spill and the implementation of a site management plan (SMP) with periodic reviews within a former MGP site. Responsibilities included field management, report writing, modeling of NAPL mobility, NAPL fingerprinting, development of a vacuum-enhanced fluid recovery (VEFR) solution, and project management (proposal and invoice submission, client communication). [Current]

FIRM

Parsons, Boston Office

YEARS OF EXPERIENCE

12

YEARS WITH PARSONS

8

EDUCATION

- MS, Geology (Structural), Western Washington University, Bellingham, WA. 2008
- BS, Geology, University of New Hampshire, Durham, NH. 2002.

PROFESSIONAL SKILLS

- Report experience: SI, RI, FS, PP, ROD, UFP-QAPP, HASP, APP/SSHP, client presentations
- 40-hour HAZWOPER certified (8-hour refresher up to date)
- First Aid/CPR/AED
- Geographical Information System (GIS) (QGIS, ArcGIS)
- EPA ProUCL Software
- SourceDK Remediation Timeframe Software
- Low-flow groundwater sampling (PFAS site lead)
- Soil sampling (discrete, CRREL 7-wheel, IS)
- Sediment/Surface water sampling
- VEFR Technique
- EM-61 MK2
- Global Positioning System (GPS/RTK) – Trimble/Garmin

PUBLICATIONS/PRESENTATIONS

- Belanger, T., 2008. Structural Geology of the Central San Juan Islands, Northwest Washington [MS Thesis]. Western Washington University, Bellingham, WA, 145 p.
- Belanger, T. and Schermer, E., 2004. Comparison of Deformation in Two Accretionary Wedge Terranes, Central San Juan Islands, WA [abs.]. Geological Society of America Abstracts with Programs, Annual Meeting, v. 38, n. 5, p. 18.

Todd Belanger (continued)

Geologist. Galena FOL, Sites SS014/SS017/SS021 Alaska. Todd provided the development of the Alaska DEC Hydrocarbon Risk Calculator (HRC) for three sites with NAPL-contaminated soil source areas. The HRC is focused on characterizing the nature, extent and risk posed by discrete, contiguous, NAPL source areas. Analysis of the HRC included the management and manipulation of a 60,000-row database leading to a refined conceptual site model and source area assessment which assisted in the selection of several site remedies included source zone excavation, bioventing, sulfate-enhanced bioremediation and free product recovery. Other duties included cleanup plan report writing. [Nov. 2015 – June 2016]

Geologist/Field Assistant for an ESTCP biogeochemical transformation demonstration using in-situ bioreactors at a site with chlorinated solvents in groundwater. Tasks included soil borings and well installation, construction oversight, and engineering systems installation. [October 2013 – March 2014]

Site Manager at an ESTCP munitions removal and geophysical classification project at Massachusetts Military Reservation. Responsible for organizing day-to-day geophysics operations, coordinating the UXO removal team and reporting to the PM. Assisted with and organized the collection of GPS, electromagnetic data (EM61) and feature attributes (e.g., orientation, item, photos). [May – June 2013]

Geologist at a former ExxonMobil site in Bayonne, NJ remedial investigation. Various field tasks including soil and groundwater sampling, soil borings, soil and groundwater characterization, well installation, well development, and well gauging. [November – December 2012; April 2013]

Geologist at Former ChevronTexaco Research Center Beacon, New York. Conducted subsurface characterization of soil and groundwater beneath former building foundations using hollow-stem auger and split spoons. Collected soil boring logs, groundwater conditions, and conducted soil sampling. [September – October 2012]

Geologist at Seneca Army Depot Activity Open Detonation Grounds Munitions Response Action. Member of the site geophysical team responsible for reacquiring points using RTK, and using EM-61, handheld GPS, and taking photos alongside a UXO dig team. [April 2012 – August 2012]

Geologist responsible for the site characterization activities at a former FAA non-directional beacon (NDB) and maintenance facility on Bimini Island, Bahamas. Tasks included soil boring and sampling, pre-demolition building material survey for hazardous materials, and characterization report writing. [October 2011]

Resident Engineer responsible for the site characterization and removal of an underground storage tank adjacent to sensitive navigation equipment at Reagan National Airport. Tasks included compliance with VADEQ regulations, observation and environmental monitoring during the tank removal, and contaminant sampling. [August 2011]

Senior Scientist/Geologist supported the Army Corps of Engineers (USACE) Military Munitions Response Program (MMRP) for site inspections (SIs) at Formerly Used Defense Sites (FUDS) and the Army National Guard MMRP SIs. In addition to extensive scientific report writing, tasks necessitated knowledge of DoD munitions and munitions constituents, geology and hydrogeology site summaries, analyzing current and historical imagery, and performing and leading fieldwork. Led two-person field team while conducting soil and water sampling at over 20 sites throughout New England. Prepared and presented briefings at public meetings and coordinates the integration of work from other employees to ensure that deliverables are on-time. Worked with EPA and state regulators to develop sampling plans, risk screening levels, and SI recommendations. [May 2008 – April 2012]

Geologist. Conducted a variety of geoengineering and environmental engineering projects for private and public entities. Tasks included slope and rock stability studies, Phase I environmental site assessment reports, septic design, engineering inspection, construction oversight, and site inspection reports. Field tasks included surface and groundwater hydrology assessments, percolation tests, soil classification, dynamic cone penetration tests, overseeing direct push and pile installation. Laboratory soils and materials testing. Construction support including earthwork observation, concrete testing and reinforcement inspection. [2005-2007]

Geologist. Provided analysis and database of geologic hazards that may impact transportation routes in NH. Collected data for input into a GIS consisting of GPS location, geology, structural data, slope stability, and slope laser profiling. Involved with thin section preparation and petrographic analysis of silica dissolution gel in concrete abutments. Participated in subsurface investigations using ground-penetrating radar (GPR) to determine bedrock depth and underground utility locations. [2002]

Appendix E

Response to Comments

Response to Comments from USEPA

Subject: Seneca Army Depot, NYSDEC Site No. 850006
Draft Perchlorate in GW – QAPP

SENECA ARMY DEPOT ACTIVITY
ROMULUS, NEW YORK

Comments Dated: 07 June 2018

Date of Comment Response: 10 October 2018

Response to Comments

Comment 1: *Worksheet #1 & 2*: Information regarding the Original QAPP that is being modified by this Addendum should be provided.

Response 1: Worksheet #1 & 2 was added to Addendum #1

Comment 2: *Worksheet #11*: Data Quality Objectives for the proposes Soil investigation should also be delineated.

Response 2: Worksheet #11 was revised to include soil.

Comment 3: *Worksheet #17:*
a. A specific reference should be provided for where in the Work Plan or QAPP is the rationale for the number and location of samples is located.

b. Since this is part of a long-term monitoring QAPP, plans for using any of the new wells for LTM of Perchlorate and 1,4 dioxane should be delineated, including the process that will be used to make this decision.

Response 3:

a. Rationale for the number and locations of samples can be found in Section 3.1 and 3.3 of the Work Plan.
b. Sampling for perchlorate and 1,4-dioxane are not part of the long-term monitoring program. They are addressing specific requests to sample these analytes by EPA and NYSDEC. While perchlorate may be associated with former activities at the OD Grounds, 1,4-Dioxane is not considered a COC at the OD Grounds.

Comment 4: *Worksheet #36*: For the electronic validation information, please provide the application that will be used, including version number.

Response 4: Electronic validation is not used. Data validation is conducted using the USEPA Region 2 SOPs for data review. Output EDDs are in Excel format workbooks. Text referencing electronic data validation was removed from Worksheet #36.

Response to Comments from NYSDEC

Subject: Seneca Army Depot, NYSDEC Site No. 850006
Draft Addendum to the Final QAPP

SENECA ARMY DEPOT ACTIVITY
ROMULUS, NEW YORK

Comments Dated: 28 June 2018

Date of Comment Response:10 October 2018

Response to Comments

Comment 1: Worksheet #11 notes, "The purpose of the perchlorate groundwater investigation is to determine the presence or absence of PFAS in groundwater as a result of ordinance detonation activities and the purpose of the 1,4-dioxane sampling is to determine the presence or absence of this compound at the Ash Landfill. Please clarify this statement.

Response 1: The second paragraph of Worksheet #11 was updated as follows:

> The purpose of the perchlorate soil and groundwater investigation is to determine the presence or absence of perchlorate in soil and groundwater as a result of ordnance detonation activities. Soil sampling locations were selected to be near the OD Hill (source) and in nearby drainage ditches. The purpose of the 1,4-dioxane groundwater sampling is to determine the presence or absence of this compound at the Ash Landfill. Additional groundwater sampling for PFAS at SEAD 25 and SEAD 26 will further delineate the area of contamination.

Comment 2: Worksheet #15 listed the 1,4-dioxane project action limit as 0.46 ug/L. The EPA's Health Advisory Level is 0.35 ug/L.

Response 2: The EPA Health Advisory Level was added to Worksheet #15 for informational purposes.

Comment 3: Résumés for the project team should be included.

Response 3: Resumes will be added to the Addendum as requested.

Comment 4: In Section ES.2, the sampling for PFAS is mentioned, but then is not addressed in worksheet #11 or #18. Worksheet #17 references Worksheet #14, which does not appear in this addendum.

Response 4: Additional information for PFAS will be addressed in the noted Worksheets and lab SOPs will be updated. Worksheet #14 was updated and added to the Addendum.